RADIATION-RESISTANT DEVICE PHENOMENA

By William C. Follmer and Dr. Arnold Berman

December 1964

Prepared Under Contract No. NASW-997 by

PHILCO CORPORATION
A Subsidiary of Ford Motor Company
Applied Research Laboratory
Blue Bell, Pennsylvania

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF APPENDIXES

SUMMARY

17886

This report describes the initial work expended with regard to setting up the equipment necessary to perform fundamental measurements on energetic electron transport through thin metal films, and the development of reactively evaporated aluminum-oxide films for use in thin-film devices.

An experiment utilizing a special low-energy electron gun as a source of energetic electrons has been set up in an ultrahigh-vacuum system and is now undergoing final tests before the start of measurements on the attenuation length of energetic electrons in thin gold films.

A majority of the facilities required for the controlled production of reactively evaporated aluminum-oxide films have been designed and constructed during the first reporting period. These facilities include a high-vacuum system and drybox assembly to prevent film contamination, a resonant quartz microscale (R.Q.M.) deposition rate monitor and controller, and an oxygen-pressure servo control system.

INTRODUCTION

The need for highly reliable solid-state electronic components for use in the space program has led to an intensive investigation of devices capable of performing active circuit functions and offering improved resistance to the radiation damage known to occur in the space environment. This investigation has shown that majority carrier devices in which the active process takes place in an extremely small volume will produce the desired improvement in radiation resistance. At present there are two approaches to such a radiation-resistant device: the metal-base transistor and the field-effect transistor. Both approaches offer the possibility of improved performance levels in addition to the desired improvement in radiation resistance. Philco has been conducting a research program aimed at developing devices of each of the above classes. Substantial progress has been made, both in the theoretical understanding of these structures and in the technology required to implement them.

The metal-base transistor (MBT) approach to a radiation-resistant device makes use of the transport of energetic electrons across thin metal films. Philco has been active in the study of such triode structures for

1

SUMMARY

17886

This report describes the initial work expended with regard to setting up the equipment necessary to perform fundamental measurements on energetic electron transport through thin metal films, and the development of reactively evaporated aluminum-oxide films for use in thin-film devices.

An experiment utilizing a special low-energy electron gun as a source of energetic electrons has been set up in an ultrahigh-vacuum system and is now undergoing final tests before the start of measurements on the attenuation length of energetic electrons in thin gold films.

A majority of the facilities required for the controlled production of reactively evaporated aluminum-oxide films have been designed and constructed during the first reporting period. These facilities include a high-vacuum system and drybox assembly to prevent film contamination, a resonant quartz microscale (R.Q.M.) deposition rate monitor and controller, and an oxygen-pressure servo control system.

INTRODUCTION

The need for highly reliable solid-state electronic components for use in the space program has led to an intensive investigation of devices capable of performing active circuit functions and offering improved resistance to the radiation damage known to occur in the space environment. This investigation has shown that majority carrier devices in which the active process takes place in an extremely small volume will produce the desired improvement in radiation resistance. At present there are two approaches to such a radiation-resistant device: the metal-base transistor and the field-effect transistor. Both approaches offer the possibility of improved performance levels in addition to the desired improvement in radiation resistance. Philco has been conducting a research program aimed at developing devices of each of the above classes. Substantial progress has been made, both in the theoretical understanding of these structures and in the technology required to implement them.

The metal-base transistor (MBT) approach to a radiation-resistant device makes use of the transport of energetic electrons across thin metal films. Philco has been active in the study of such triode structures for

1

several years, and a comprehensive theory for the performance of these devices has been developed. Fundamental measurements programs have been under way for some time to determine the attenuation length of energetic electrons in various metals and also to evaluate the collection efficiency of various collector structures.

Studies are also being conducted to develop the needed technology for the production of very thin, compact, insulating films to be used in active thin-film devices. Two such methods are known; namely, gaseous anodization[1] and reactive evaporation.[2,3] Both of these processes are capable of producing the desired film dimensions. The former method has the disadvantage that such films can be produced only on valve metals such as aluminum, tantalum, etc. It is, however, reproducible and uniform. The latter method, with which Philco has had considerable success in recent months, is theoretically applicable to film deposition on any metal or insulator, although the work done to date has been limited to aluminum substrates. While this method shows promise of also being extremely reproducible, more studies are necessary to prove this point.

Once the proper techniques have been developed for the production of these thin insulating films, it is necessary to evaluate their use in thin-film active devices. This will be done as part of Philco's fundamental measurements program. In this way, complete characterization of the individual film elements to be used in thin-film active devices can be obtained so that the experimental results obtained on triode structures can be better evaluated and understood.

ELECTRON TRANSPORT THROUGH THIN FILMS

The objective of this part of the contract is to study the transport of energetic electrons across thin metal films, and the collection of these electrons. Under a previous program, Philco has conducted extensive fundamental studies regarding the mean free path of energetic electrons in thin films, and the collection efficiency of metal-insulator and metal-semiconductor interfaces. Results of these theoretical studies have been reported.[4] They show that the problems of transport and collection are the fundamental processes that are most important if one is to successfully operate a metal-base transistor. This preliminary work has also suggested an experimental technique which should provide information regarding both the mean free path and the collection efficiency. It is this experiment which will be pursued during the course of this contract.

Experimental Studies of Energetic Electrons

It is desired to examine the transport of a directed beam of fast electrons through a thin metal film and their collection at a metal-semiconductor barrier. To make this study a gold-silicon diode will be bombarded by an external beam of low-energy electrons. The diode will act in a way similar to the collector element of a metal-base transistor, but the use of an external electron beam will avoid the ambiguities of interpretation which beset an all-thin-film device.

An energy diagram of the experiment is shown in Figure 1. The electrons emitted from the tungsten cathode will have an energy $\mathcal{E}$ above the Fermi level of the gold film. The minimum value of $\mathcal{E}$ will be determined by the gold work function ϕ_2. This can be reduced to about 1.8 volts by a monomolecular layer of BaO. Upon entering the gold film, the electrons will acquire an additional energy of several volts, with momentum directed normal to the film. Most of this kinetic energy will be lost upon entering the conduction band of the silicon. The very small critical angle of collection will ensure that any collision in the gold base layer will prevent collection. Those electrons that do not collide in the gold film and do not back-scatter from the silicon will be observed as a collection current. The remaining electrons will return to the cathode through the base layer. By measuring the fraction collected as a function of film thickness, the total mean free path can be determined and the transport efficiency separated from the effects of collection efficiency. This will be done over a range of electron energies which can hopefully be extrapolated to the low-energy limit.

3

LEGEND:

ϕ_1: WORK FUNCTION OF TUNGSTEN.

ϕ_2: REDUCED WORK FUNCTION OF GOLD.
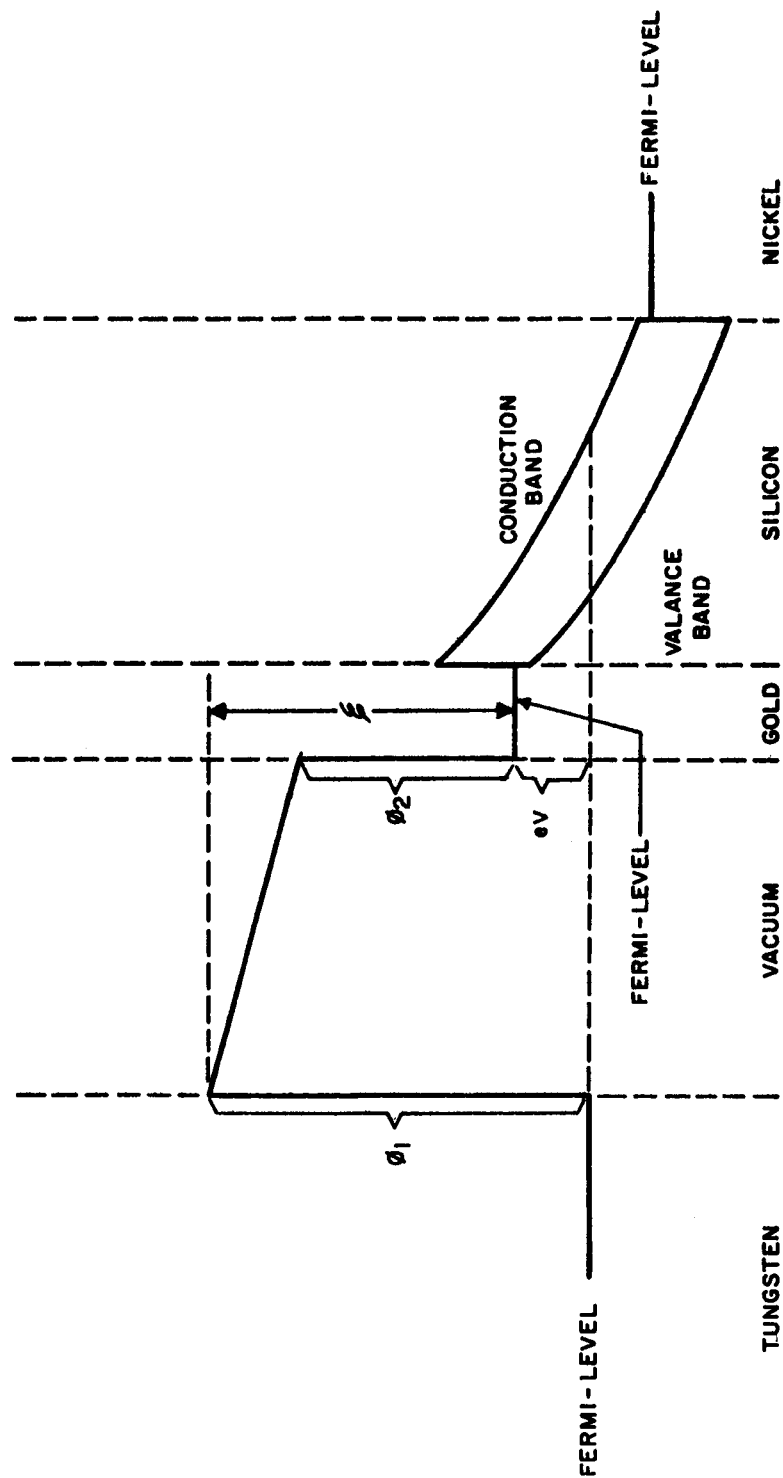
$\xi = \phi_1 - ev$

Figure 1. Energy Diagram of Electron Transport Experiment

4

When the details of this experiment have been worked out, it can be used as a tool for measuring the transport efficiency of any metal base and the collection efficiency of any interface.

Experimental Details

Electron gun. - Because of the requirement of a very-low-energy electron beam, the space charge will severely limit the magnitude of the beam current available. To partially compensate for this limitation, both the cathode and diode areas are made large, about one cm^2. The cathode disk is heated by electron bombardment at high voltage from the rear. This allows heating by a relatively small current so that large voltage drops do not develop along the cathode and make the electron energy uncertain.

The electron energy ε is known from the tungsten work function and the applied voltage. The cathode may be heated to 2000°C to expose a clean surface whose work function is well known. The experiment is carried out at pressures of about 10^{-8} torr to keep the surface clean. The gun has gone through various stages of design during this experiment. Figure 2 shows schematically the present design which was assembled and tested during the past quarter. A photograph of the actual gun is shown in Figure 3.

The cathode is separated from the sample by about 2 inches to prevent heating of the sample. The diameter of the gun must be correspondingly large to transmit the electron beam. This separation results in severe space-charge limitation of the beam current. To circumvent this condition, an accelerating grid is mounted at the end of the gun and the space charge is confined to the small grid-sample separation.

The shield, which is closely spaced to the cathode, prevents electrons from the tantalum cathode holder, as well as ions created in the vicinity of the filament, from entering the cathode beam. These ions, which manifested themselves in a reverse current at retarding potentials, plagued earlier gun designs.

The focusing electrode serves to confine the beam to the incident side of the diode. This is important, since it is expected that collection efficiencies as small as 10^{-4} will be measured. The beam current displays a sharp maximum as the focusing voltage, V_F, is varied. This makes a convenient control to modulate the beam current.
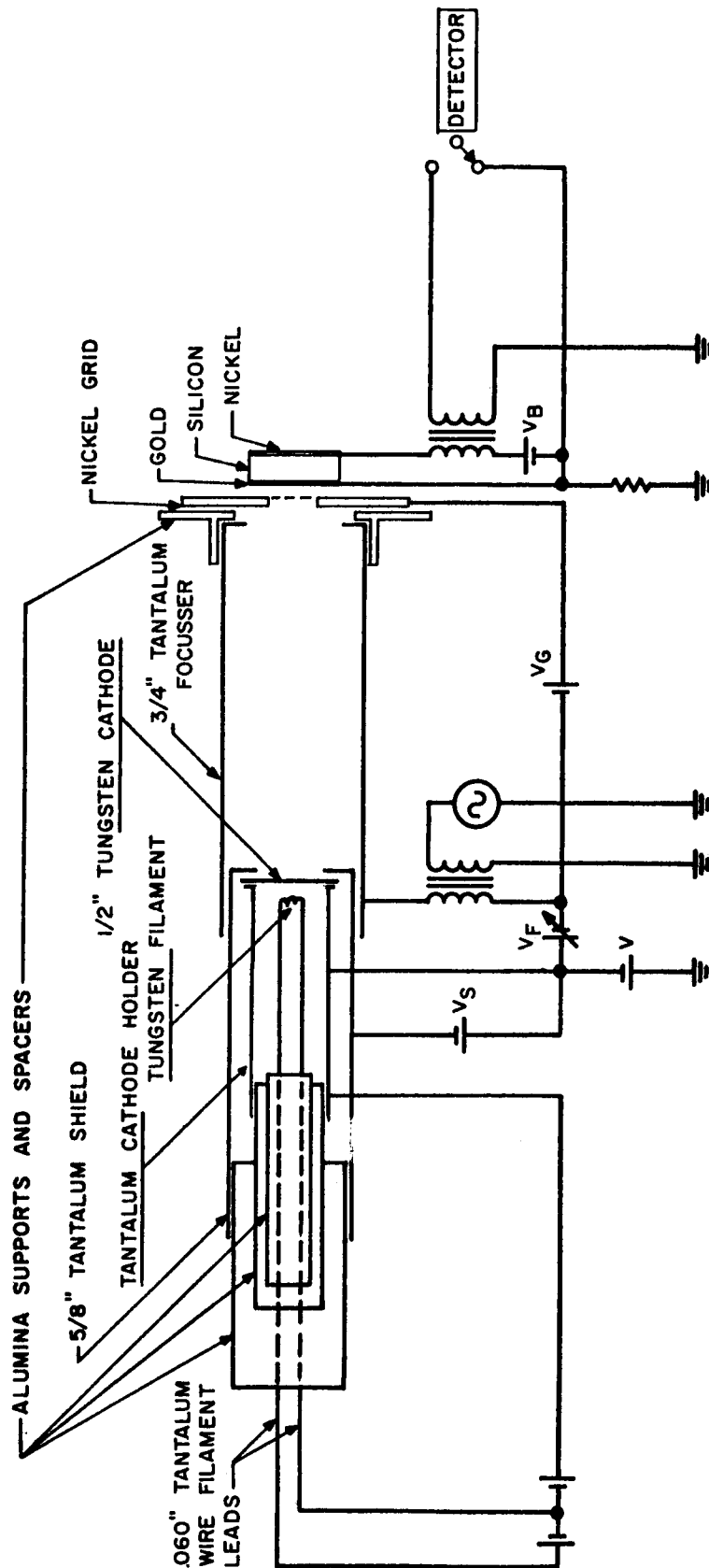
5

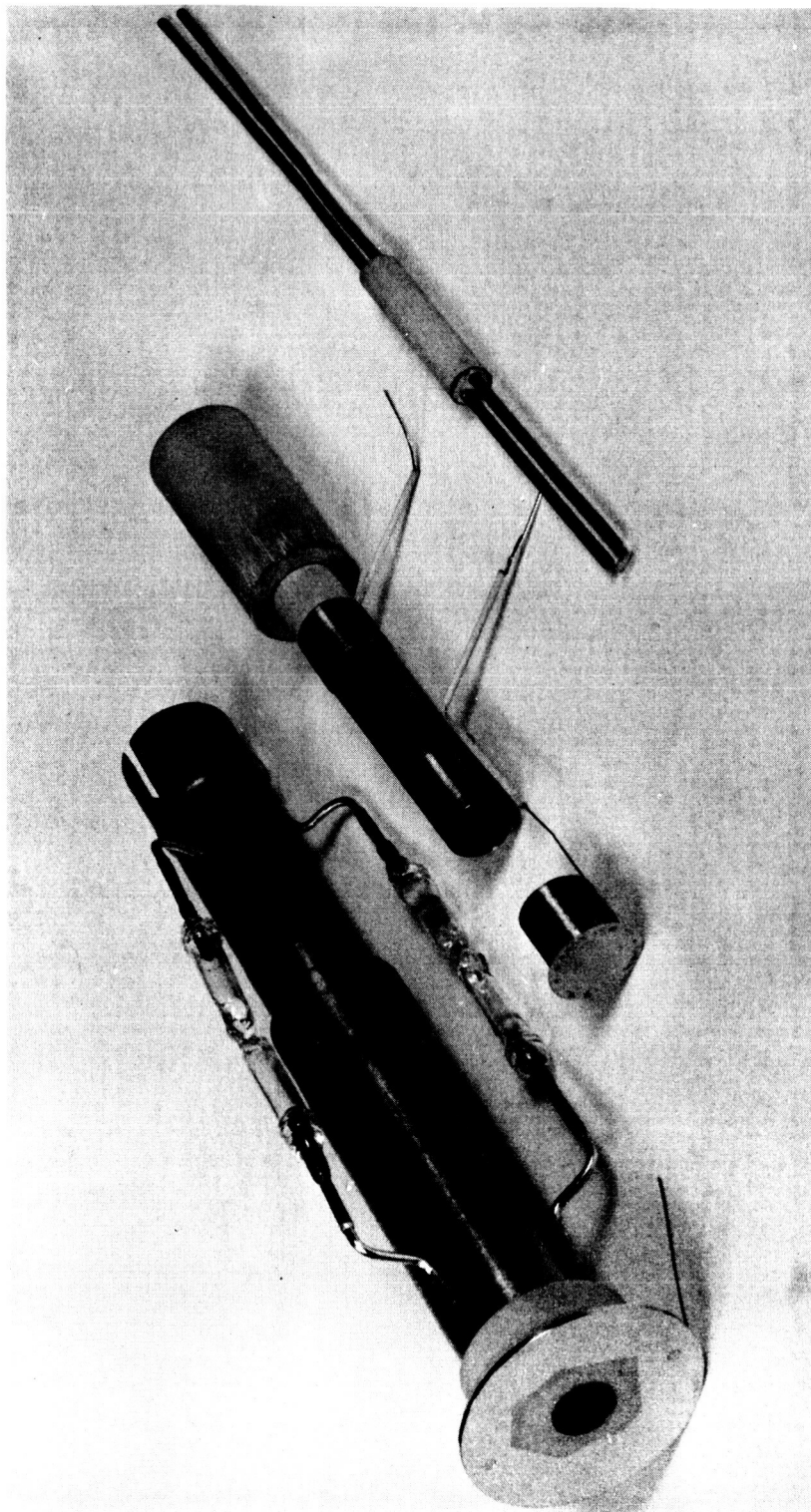Figure 2. Simplified Experimental Setup for Electron Transport Measurements

6

Figure 3.   Low-Energy Electron Gun

Current-voltage characteristics are shown in Figure 4 for various values of grid voltage V_G. In these curves, V_F and V_s are optimized for a maximum beam current.

Diodes. - The diodes are made by evaporating a gold film onto a silicon blank which has been prepared as shown in Figure 5. Gold was chosen for the initial work because of its chemical stability, known phonon mean free path, and anticipated electron-electron mean free path, and because of previous work done on reducing the gold work function with barium oxide. A resonant quartz microscale (R.Q.M.) will be used to monitor the mass per unit area of both the vacuum-deposited gold film and the barium-oxide monolayer.

The nickel makes ohmic contact to the silicon; the gold layer provides contact to the gold film to be evaporated onto the exposed silicon; and the oxide layer insulates the gold contact layer from the silicon.

A number of silicon blanks were prepared during the reporting periods, but they proved to have intermittent electrical shorts through the oxide layer. Another group is now being prepared under more careful conditions to prevent the reoccurrence of this problem.

Circuitry. - In order to measure collection currents as small as 10^{-11} ampere, sensitive detection methods and a high degree of isolation from spurious coupling are required. The grid, which is kept at a low impedance to ground, serves to capacitively decouple the diode from the focuser and thus prevent stray currents in the absence of a beam current.

The circuit shown in Figure 2 has been assembled and preliminary tests made. With narrow banding, the small currents can be measured with the PARJB5 detector.

Figure 4. Current-Voltage Characteristics of Low-Energy Electron Gun

LEGEND:

$T_C = 1375°C$
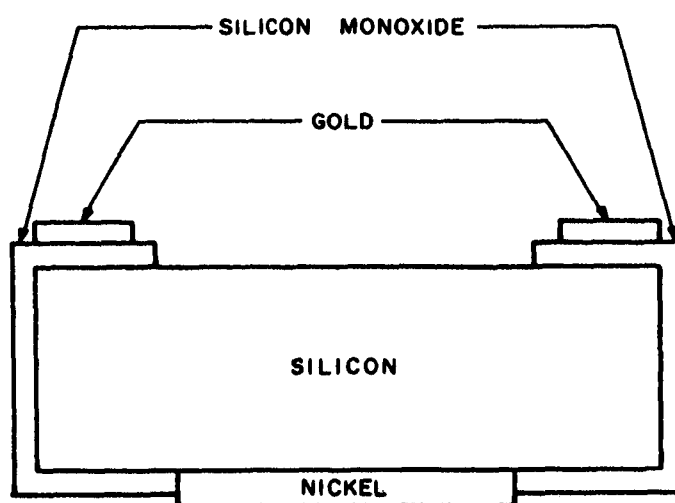
$V_F = 0.5$ VOLTS

$V_S = 0$

9

Figure 5. Collector Diode

REACTIVELY DEPOSITED ALUMINUM OXIDE

Reactive Evaporation

The high resistivity, high breakdown strength, and stability of aluminum oxide have encouraged its investigation as a possible candidate for a thin-film insulator. Early work on aluminum oxide utilized the natural oxide as an insulating barrier. This technique did not provide a reproducible, high-field insulator. There are at least two techniques that have been shown to produce excellent insulating films. These are the gaseous anodization[1] of a previously deposited film and the reactive evaporation of aluminum oxide.[2,3] The reactive evaporation technique has the advantage in that the insulating film can be deposited upon any desired substrate, and for this reason is being studied for application to thin-film active-device fabrication.

To obtain reactively evaporated aluminum films, pure aluminum is evaporated in a background pressure of oxygen of approximately 1×10^{-3} mm. The deposition rate is purposely kept low so that the aluminum deposited may be continuously oxidized by the oxygen present in the system. A typical Al_2O_3 deposition rate for the initial work was 24×10^{-9} grams per cm^2 per second. This corresponds to 3×10^{14} aluminum atoms per second per square cm. At an oxygen pressure of 10^{-3} mm, one can calculate that the resulting oxygen molecule impact rate is 6×10^{15} impacts per cm^2 per second. It can also be calculated[4] that the mean free path of the evaporated aluminum is of the order of 8 centimeters at 10^{-3} mm oxygen. With a source-to-substrate spacing of less than the mean free path, it is then reasonable to assume that the majority of the oxidation occurs after the aluminum atoms have condensed on the substrate. It can be seen from these calculations that the degree of oxidation in the deposited film will depend upon the oxygen pressure and the deposition rate. Previous experimental work has shown that films of resistivity varying from essentially that of pure aluminum, 10^{-5} ohm per cm, to that of aluminum oxide, 10^{+14} ohms per cm, can be prepared by varying the oxygen pressure and/or the deposition rate. Thus, the requirement exists for tight control of the deposition rate and oxygen pressure if one desires films of known and controllable composition.

In addition to these problems in preparation, aluminum oxide absorbs a substantial amount of water vapor if exposed to a wet gas. This water-vapor absorption could seriously affect the electrical characteristics

of the insulating film. It is therefore important that the films be protected from contamination. Further, as in any film work, dust contamination is a serious problem and must be eliminated. These considerations have led to the development of the following experimental procedure for the study of reactively evaporated films of aluminum oxide.

Deposition Technique

The preparation of aluminum-oxide samples for electrical evaluation will consist of three major steps. In the first step, metal electrodes will be evaporated onto the glass substrates, using conventional metal evaporation techniques and contact masks to obtain the desired patterns.

The glass substrates with the previously deposited metal electrodes will then be placed in a second mask and re-installed in the vacuum system. Pure aluminum metal will be evaporated from a tungsten boat in the vacuum system that has previously been evacuated to below 10^{-6} mm Hg and then back-filled to approximately 10^{-3} mm Hg with oxygen. During evaporation, while oxygen continuously flows through the system, the servo-control leak will maintain the pressure at the preset level.

The resulting film of aluminum oxide is deposited through the aluminum contact mask onto the glass substrates. The R.Q.M. sample oscillator mounted adjacent to the substrates also receives a film deposit. This deposit is monitored and provides the signals that are used to control the deposition rate.

Finally, counterelectrodes are applied to the aluminum oxide by a second metal evaporation.

The glass substrates will then be scribed and the individual samples mounted in TO-5 headers for electrical evaluation. During the encapsulation procedure the samples will be maintained in a dry atmosphere of nitrogen gas.

Electrical Evaluation

Initially, films will be measured for resistivity as a function of temperature, dielectric strength, and dielectric constant.

Resistivity will be obtained from a four-terminal resistance measurement and from multiple-beam interferometer thickness measurements. The thickness measurements will be correlated with the R.Q.M. mass measurements to obtain an indication of the film density.

A low-frequency capacity bridge (100 kc) will provide semiprecision measurements of the capacity for dielectric-constant calculations. Capacity-type samples will also be utilized in the measurement of breakdown voltage.

Facilities for the Production of
Reactively Evaporated Aluminum-Oxide Films

Practically the entire effort during this reporting period was devoted to procuring, designing, and assembling the necessary facilities required for the production of reactively evaporated aluminum-oxide films. The following paragraphs summarize these facilities and their present status. Detailed descriptions of these facilities are included as Appendixes A, B, and C.

Vacuum system and drybox assembly. - The aluminum-oxide films will be prepared in an oil-diffusion pumped-vacuum system capable of ultimate pressures of 3×10^{-7} mm. This system utilizes a liquid-nitrogen cooled cold trap to prevent back-streaming of the pumping oil. A drybox unit has been received and is now being installed on the vacuum system to eliminate the water-vapor contamination of the prepared films. This drybox unit will be continuously flushed with nitrogen gas obtained from the boil-off of liquid nitrogen. A detailed description of this assembly is given in Appendix A.

To further reduce the possibility of dust contamination, an absolute filter and recirculating system is planned for installation on the drybox.

The vacuum-system/drybox assembly is mechanically complete, but the electrical circuits and gas feeds are yet to be installed.

Oxygen-pressure monitor and control system. - The oxygen-pressure monitor and control system utilizes a commercial ionization gauge, Veeco R31-X, as the pressure-monitoring instrument. The recorder output of this instrument is coupled to the pressure-controller electronics section which provides the necessary signals to regulate the oxygen pressure in the system with a servo-driven leak valve. A Leeds and Northrup chart

recorder is also connected to the recorder output of the pressure gauge to provide a continuous record of the system pressure. The oxygen-pressure control system is provided with interlocks to prevent inappropriate opening of the leak valve. The ionization gauge, pressure-control electronics, and chart recorder are mounted in a standard relay rack located adjacent to the vacuum system. A detailed description of this assembly is contained in Appendix B.

This unit is completed and will be tested upon completion of the vacuum system and drybox assembly.

Deposition controller and mass monitor. - The deposition controller and mass monitor utilizes the shift in resonant frequency of a quartz crystal when a sample of the film is deposited on the surface of the crystal.[5,6,7] The shift in resonant frequency of the crystal has been shown to be proportional to the mass per unit area of the deposited film. This frequency shift is monitored and provides the information on the deposited mass which is proportional to film thickness, assuming that the density is constant. This mass signal is also differentiated and provides the deposition-rate signal. A servo system compares the deposition-rate signal to a reference signal and adjusts the evaporator temperature via a servo-motor-driven variac to obtain the desired rate. A detailed description of the resonant quartz microscale (R.Q.M.) deposition-rate controller can be found in Appendix C. This equipment is at the present time completely designed and about 95 percent constructed, awaiting only the delivery of some small components to allow completion. The deposition-rate controller and mass-monitor components are mounted in a second relay rack which will be located adjacent to the vacuum system.

A two-channel strip-chart recorder is needed to provide continuous records of deposition rate and deposited mass. Such a unit has been on order for some time.

Vacuum system accessories. - A water-cooled support for the in-vacuum components has been designed and the commercial elements ordered. This unit will provide a single mounting post to which the R.Q.M. sample oscillator, substrate holder, substrate temperature monitor, and shutter will be mounted. This single post will offer a maximum of versatility while occupying a minimum volume in the vacuum system.

Initial sample masking. - A set of three masks has been designed and is now in drafting for detailed design which will provide samples suitable for

14

resistivity, dielectric constant, breakdown-field, and high-field mobility measurements. Figure 6, parts A, B, and C, are sketches of the preliminary mask design. Aluminum has been chosen as the mask material because of its ease of machinability.

Substrate material. - Previous work in vacuum-deposited thin films has indicated that microscope slide cover glass, Corning type No. 2, has very smooth surfaces and is suitable for the deposition and study of thin films. In addition, these glass substrates are easily scribed and broken into individual samples which are suitable for encapsulation using standard transistor packages. This encapsulation will provide further protection from contamination while the films are characterized.
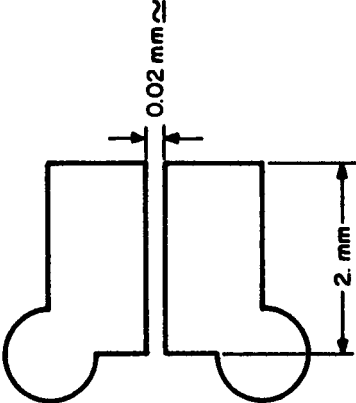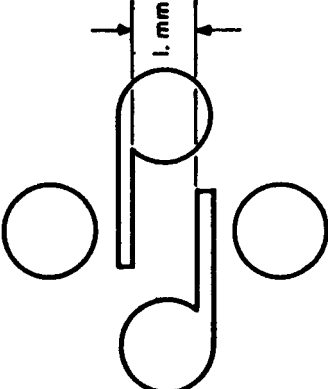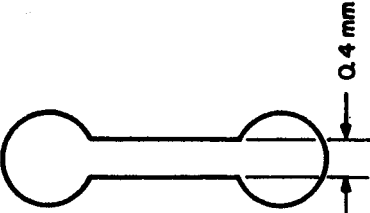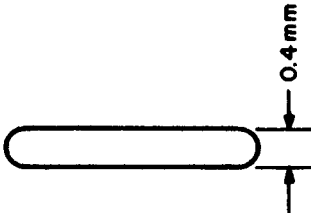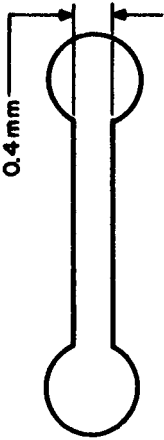
Figure 6. Typical Mask Patterns

16

APPENDIX A

VACUUM SYSTEM AND DRYBOX ASSEMBLY

Vacuum-System Components

A Kinney KC 15 mechanical pump is used as the roughing pump and backing pump for a CVC PMCU-721 oil-diffusion pump. The diffusion pump is connected through a CVC BCN liquid-nitrogen-cooled chevron cold trap and a Temascal high-vacuum valve to the modified 16-inch Kinney baseplate. The baseplate unit now contains thirteen 3/4-inch nominal diameter holes through which the vacuum-system accessories will be installed.

Veeco SV62S solenoid valves are used for roughing and backing. Initially, a 14-inch-diameter glass bell jar will be used. A counterbalanced, motor-driven, bell-jar hoist completes the vacuum-system installation. The electrical and gas-feed connections are shown in Figures A-1 and A-2.

The Drybox Unit

A specially designed plexiglass drybox (Hydrovoid) has been obtained from Air Control. This unit provides both a controlled atmosphere and protection from dust contamination for the prepared samples. The bell-jar unit is sealed with a flexible bellows to allow raising the bell jar. The sliding hand-port assembly incorporated into this unit further increases its versatility.

Nitrogen gas, obtained from the evaporation of liquid nitrogen, is continuously fed into the drybox through a pressure regulator to maintain a positive internal pressure. A needle-valve-and-flow-meter unit mounted on the vent line provides both monitoring and control of the gas-flow rate.

A future addition to the system will be an absolute filter in a recirculating system to remove any remaining dust in the drybox. Details of the gas feed are shown in Figure A-2 referred to previously. Figure A-3 is a photograph of the complete vacuum system and drybox assembly.
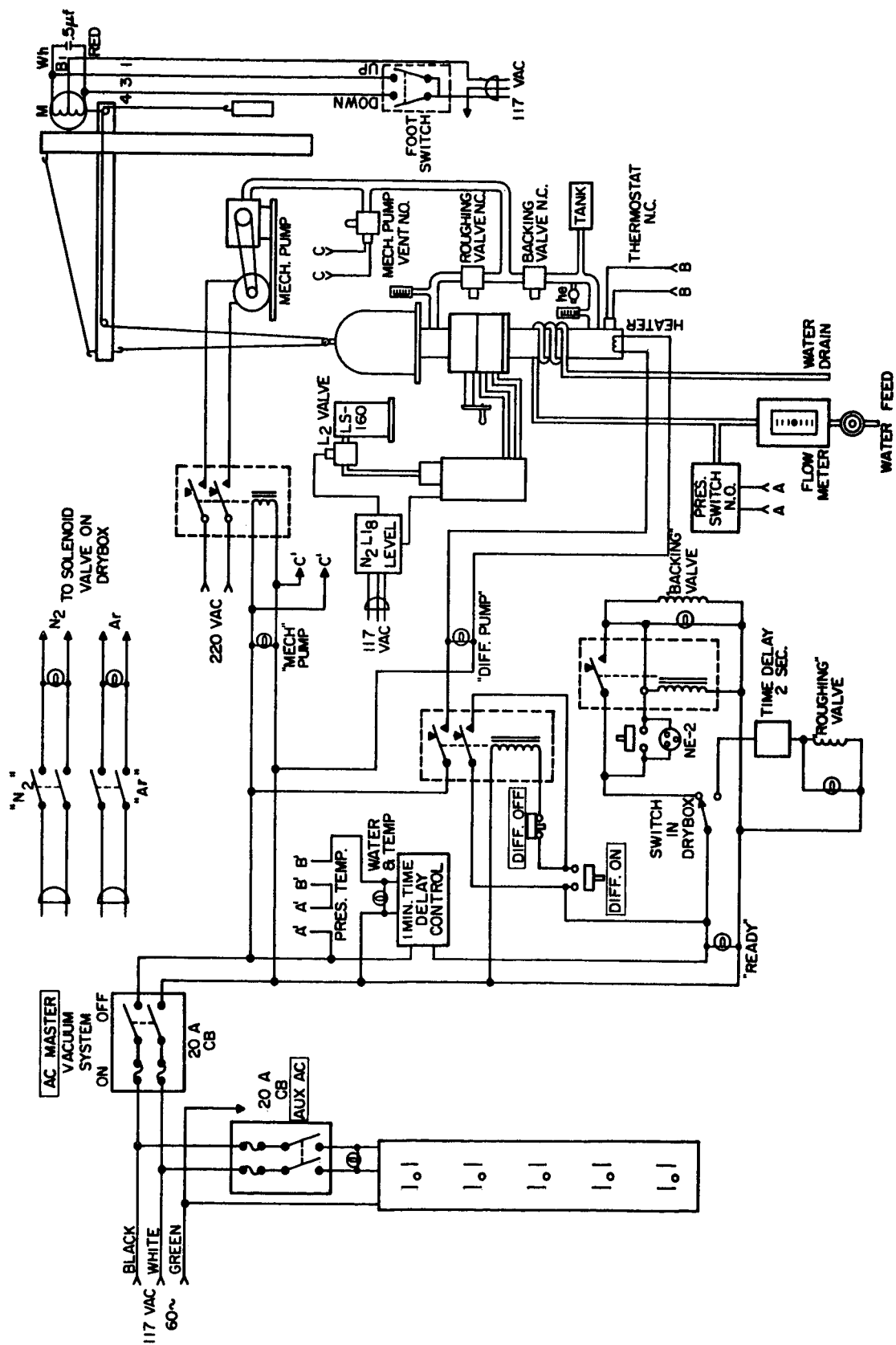
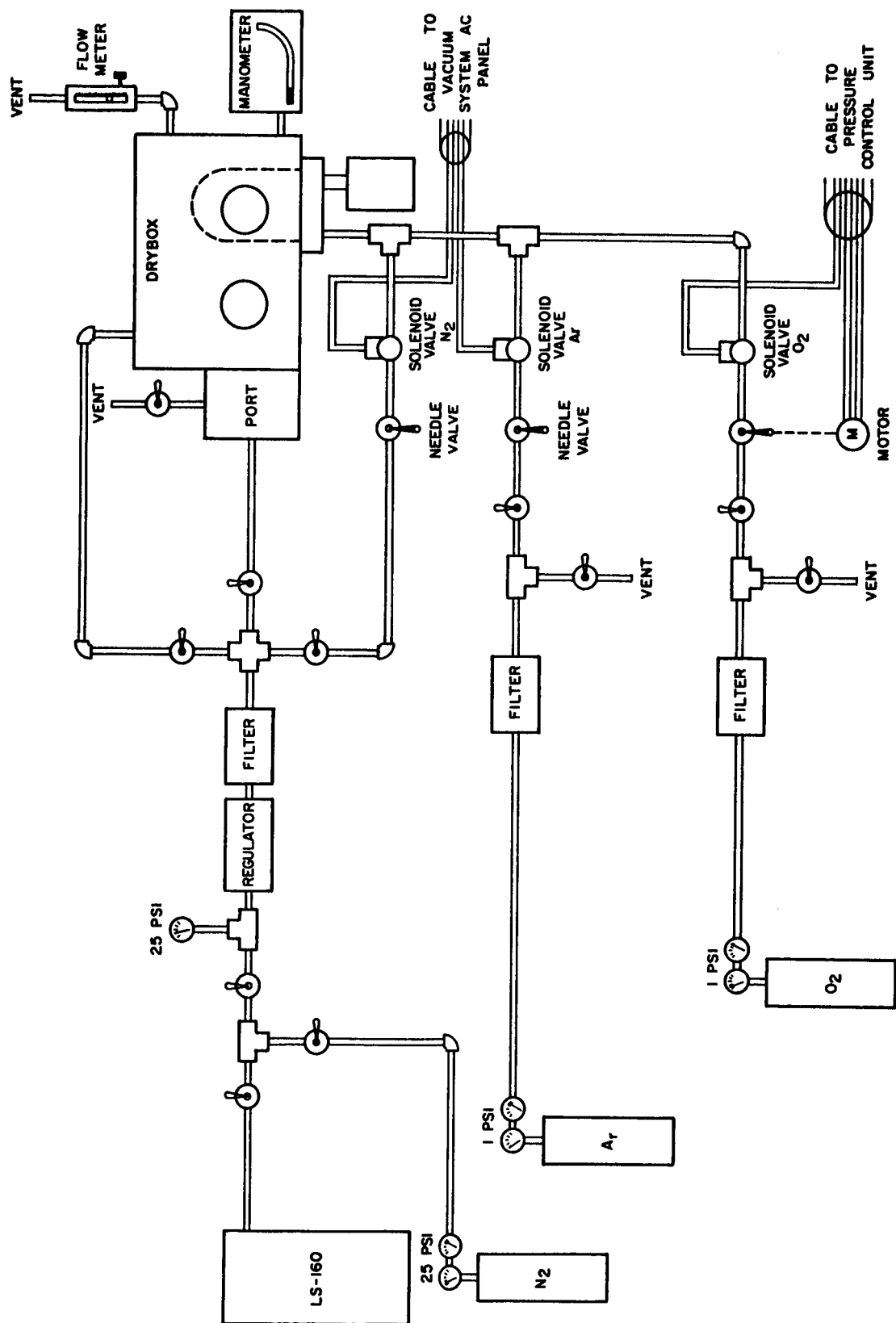Figure A-1. Vacuum System AC Schematic

18

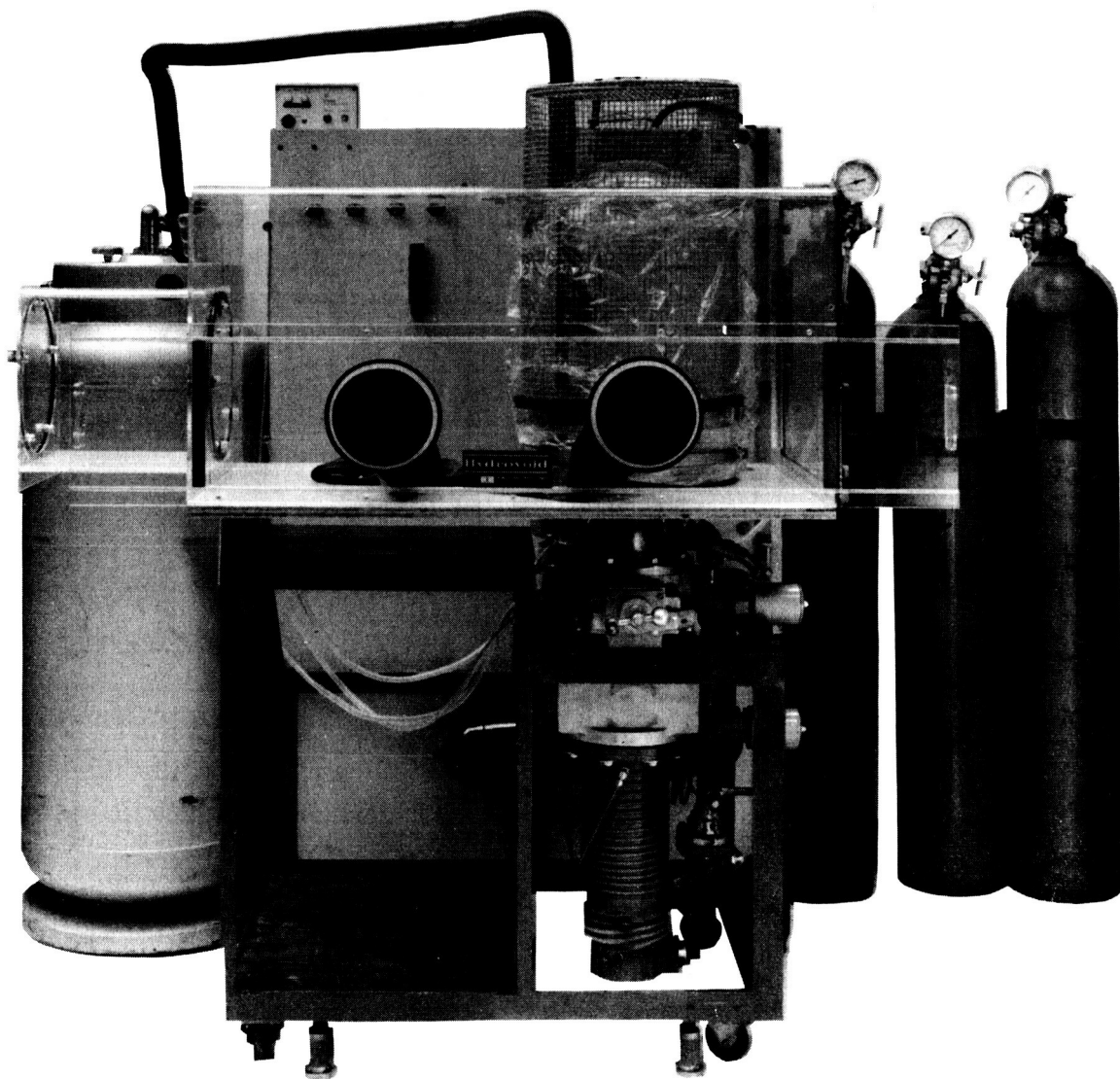Figure A-2. Gas Feed for Vacuum System and Drybox

19

Figure A-3. Vacuum System and Drybox

APPENDIX B

PRESSURE MONITOR AND OXYGEN-PRESSURE CONTROL SYSTEM

The Pressure Monitor

A Veeco ionization gauge (RG-31-X), covering the pressure range from 10^{-3} to 10^{-11} mm mercury, is the basic pressure-sensing element. This unit also provides a recorder output of 0 to -2 volts, which is proportional to the pressure being measured.

Two Veeco thermocouple gauges, one on the roughing line and one on the backing line, provide pressure indication in the range from 1 to 1000 microns.

A Leeds and Northrup model H strip-chart recorder is connected through an attenuator to the recorder network of the ionization-gauge unit and provides continuous records of system pressure.

Pressure Controller

The pressure controller utilizes the recorder output of the pressure-gauge unit. The output voltage is attenuated and compared to a reference voltage. The resulting error voltage is amplified in a Honeywell type 356413-3 60-cycle servo amplifier which then drives a servo motor. The servo motor is coupled to a leak-valve unit mounted on the vacuum-system baseplate. Any error in pressure produces an increase or decrease in the leak-valve setting and thereby holds the pressure at the established level. A block diagram of the pressure-control system is shown in Figure B-1, and a detailed schematic diagram is shown in Figure B-2. A parts list for the pressure controller is on the page following Figure B-2. Figure B-3 is a photograph of the complete control chassis.

The leak-valve unit utilizes a Nupro needle valve driven by a Borg servo motor. Detailed drawings of this unit are shown in Figures B-4, B-5, and B-6; Figure B-7 is a photo of the assembled valve. The parts list for the servo-driven leak valve follows Figure B-4.

The pressure monitor and pressure controller are installed in a standard 6-foot relay rack which is located adjacent to the vacuum system. Figure B-8 is a photograph of the assembled unit.
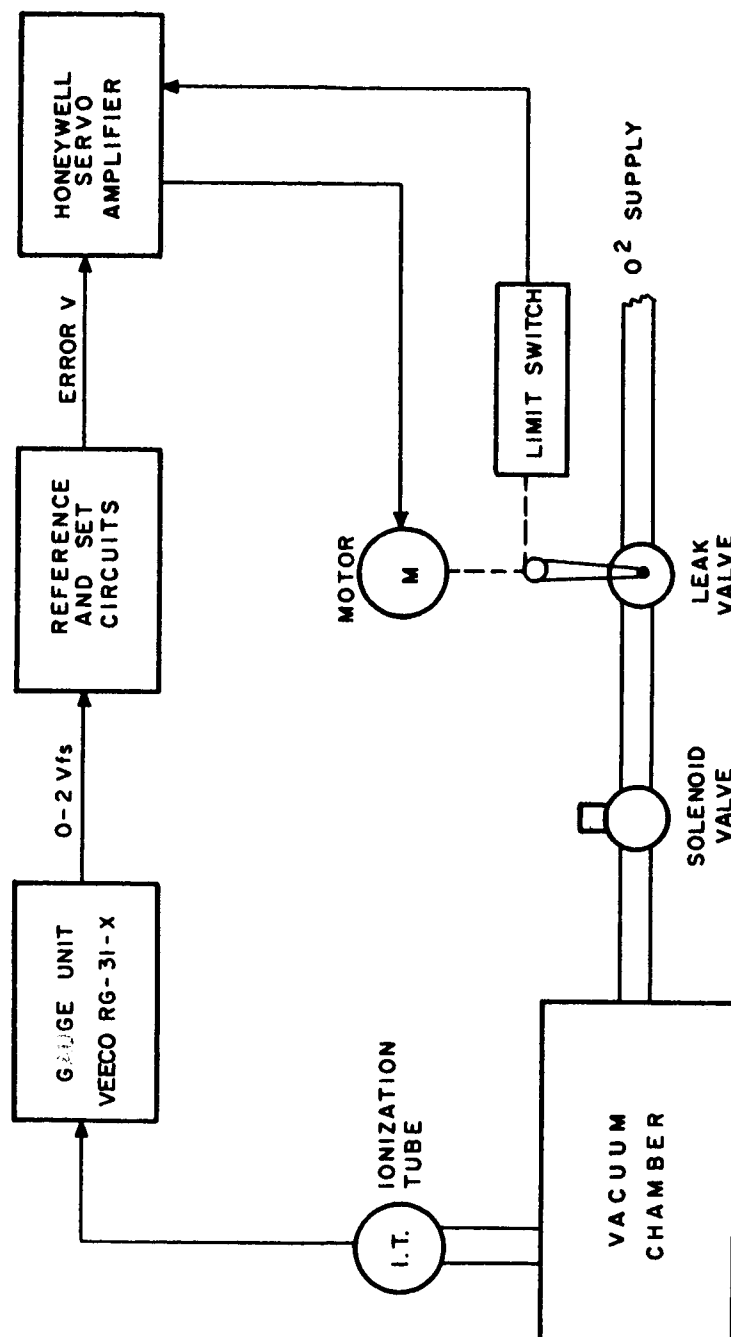
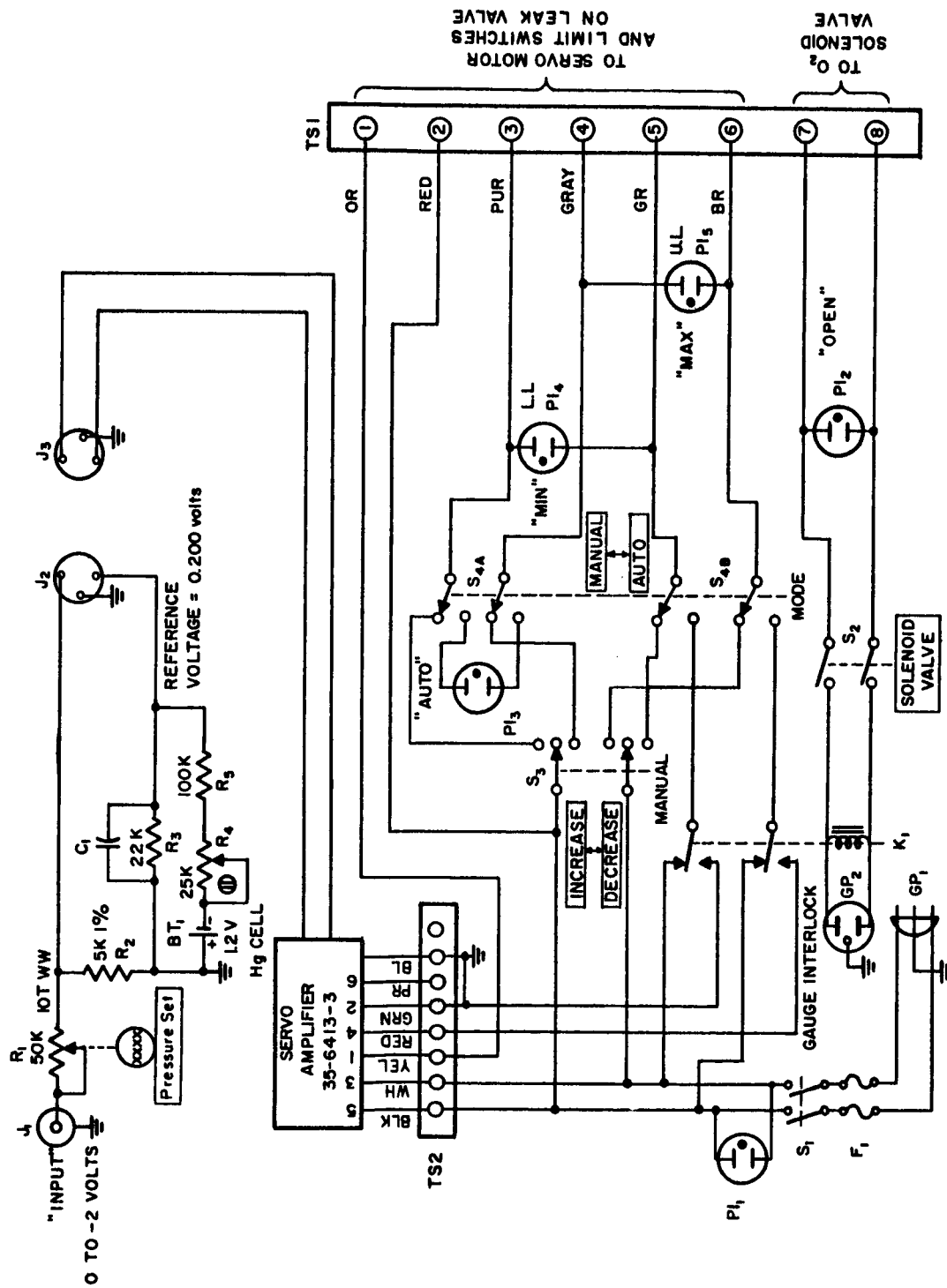Figure B-1. Pressure-Control System, Block Diagram

Figure B-2. Pressure Controller, Schematic Diagram

Parts List for Pressure Controller
(Figure B-2)

Symbol	Description
R_1	50K, potentiometer, 5%, 10-turn, W.W, Borg No. 2201B
R_2	5K, 1%, film resistor, 1/2 watt
R_3	22K, 10%, carbon resistor, 1/2 watt
R_4	25K, potentiometer, 2 watt
R_5	100K, 10%, 1/2 watt, carbon resistor
C_1	Select to provide proper damping
BT_1	1.2-volt mercury battery, Mallory No. RM-42R
SA_1	Servo amp., Honeywell No. 356413-3
J_1	BNC, UG-1094/U
J_2	Connector, audio, Amphenol No. 91-854
J_3	Connector, audio, Amphenol No. 91-856
G_{P1}	Parallel ground plug, male
G_{P2}	Parallel ground plug, female, chassis
F_1	Fuse, 2-ampere
S_1, S_2	Switch, DPST, Cutler Hammer No. 8823K5
S_3	Switch, DPDT, Cutler Hammer No. 8837K4
$S_{4A,B}$	Switch, 4PDT, Cutler Hammer No. 8838K4
Pl_1, Pl_2	Pilot light, Dialco 931
Pl_3, Pl_4, Pl_5	Indicator lights, Drake No. 110-022 (Hi Brite)
T_{S1}, T_{S2}	Terminal strip, Cinch-Jones, 8-terminal
K_1	Relay, Potter Brumfield, KL11A

24
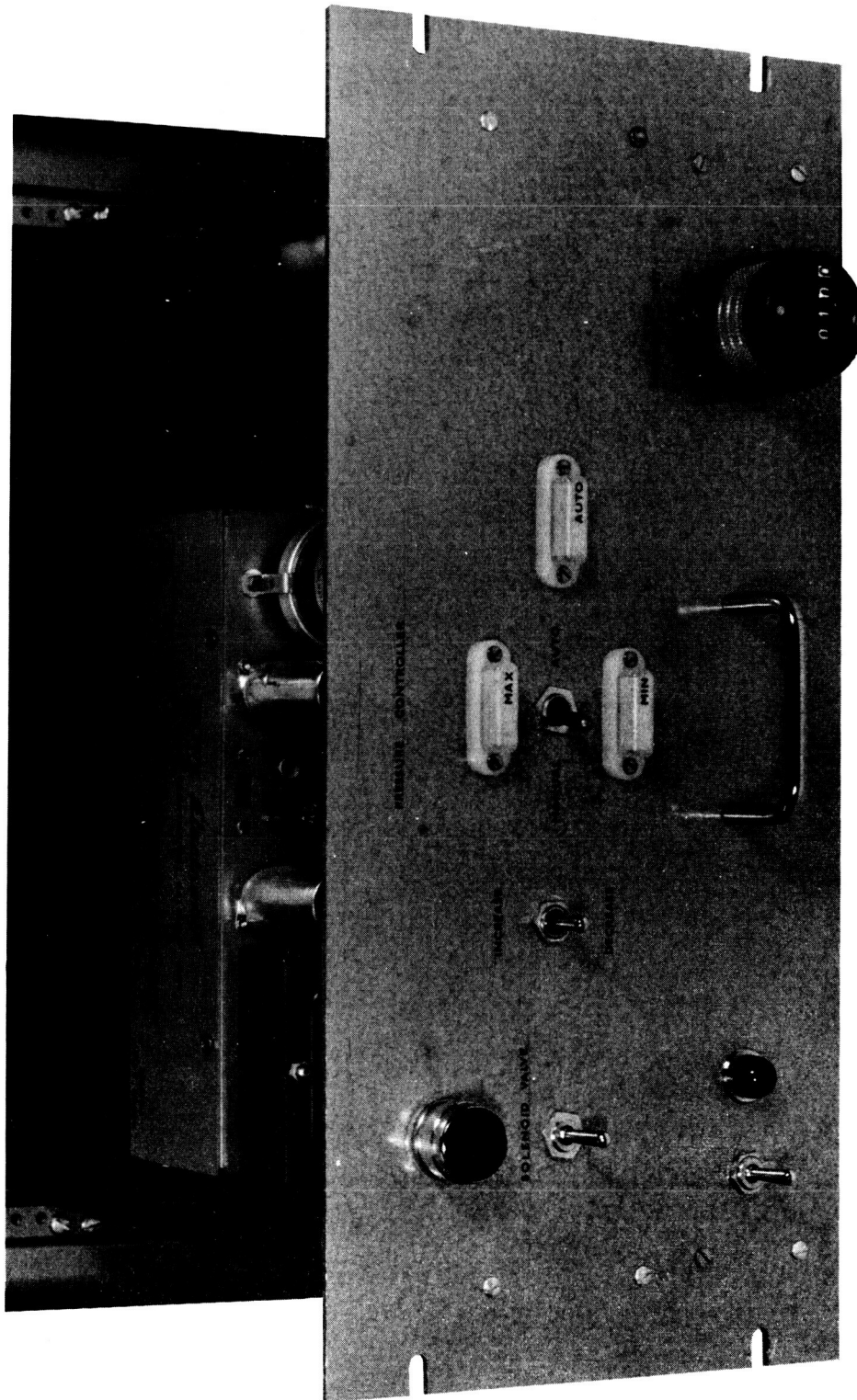
Figure B-3. Pressure Controller

25
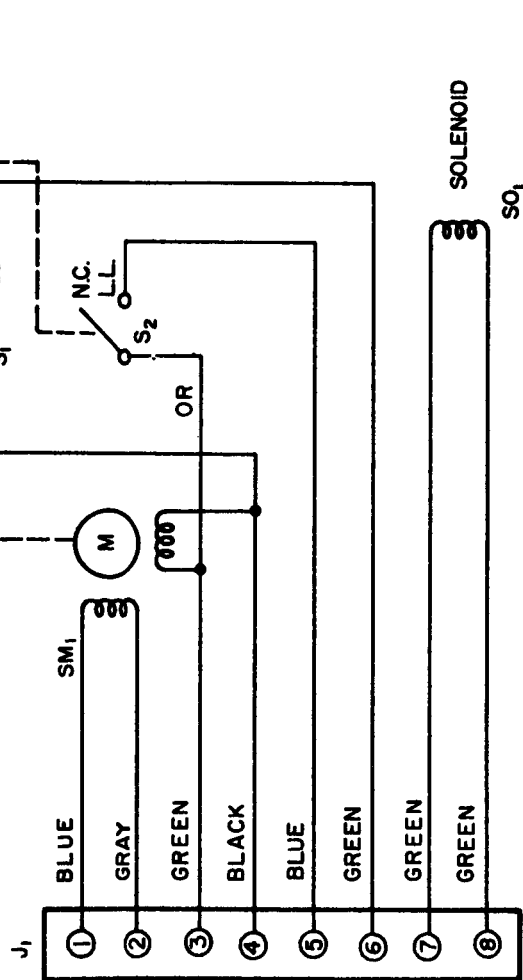
Figure B-4. Servo-Driven Leak Valve, Pressure Control System

26

Parts List for Servo-Driven Leak Valve
(Figure B-4)

Symbol	Description
J_1	Female Socket, Cinch-Jones, 8-terminal S308
SM_1	Servo motor, Borg No. 1062
S_1	Subminiature basic switch, microswitch No. 1SX1-T (MS24547-1)
S_2	Subminiature basic switch, microswitch No. 1SX1-T (MS24547-1)
SO_1	Solenoid valve, Hoke B90A380G
SL_1	Slip clutch coupling, Northfield Precision Instrument No. LCC-33
Bl_1	Bellows coupling, Sterling No. G404-34
LV_1	Metering valve, Nupro No. B252

Figure B-5. Servo-Driven Leak Valve Details

28

Figure B-6. Servo-Driven Leak Valve, Assembly View

Figure B-7. Servo Leak Value

Figure B-8.   Pressure Monitor and Controller

the oscillation frequency and provide a reading proportional to the frequency shift, which is the deposited mass signal. Note that the sensitivity of the R. Q. M. is a function of the normal operating frequency.

The Deposition-Control System

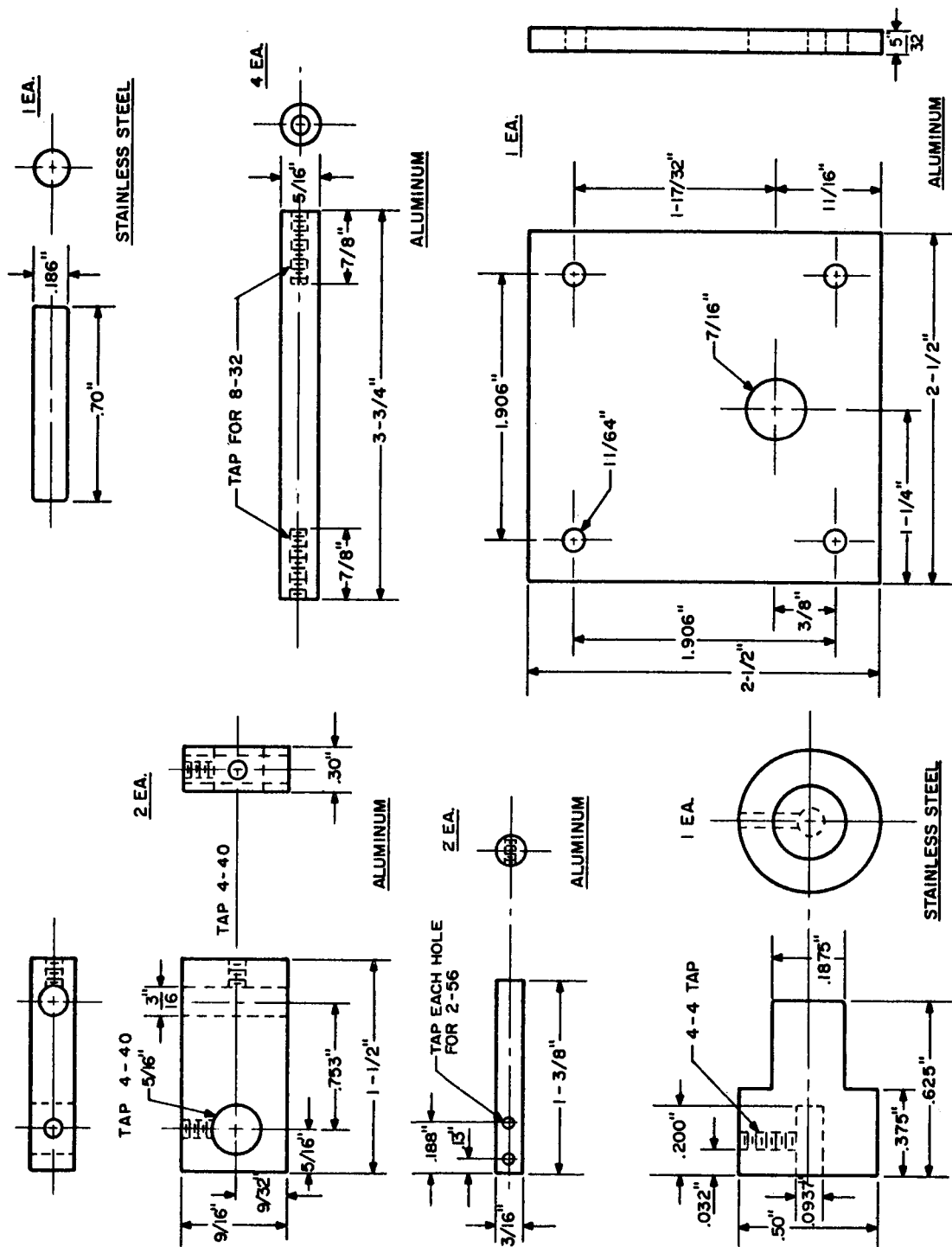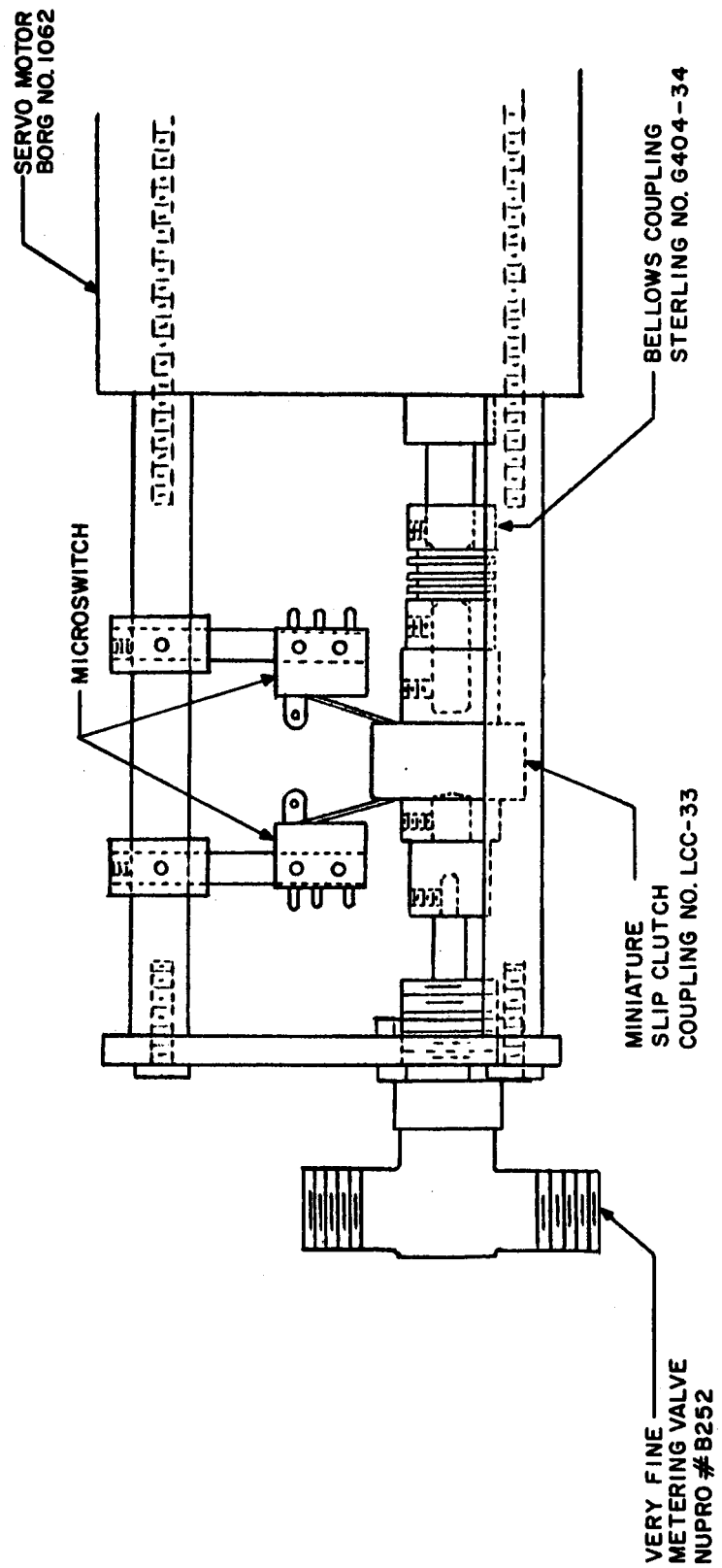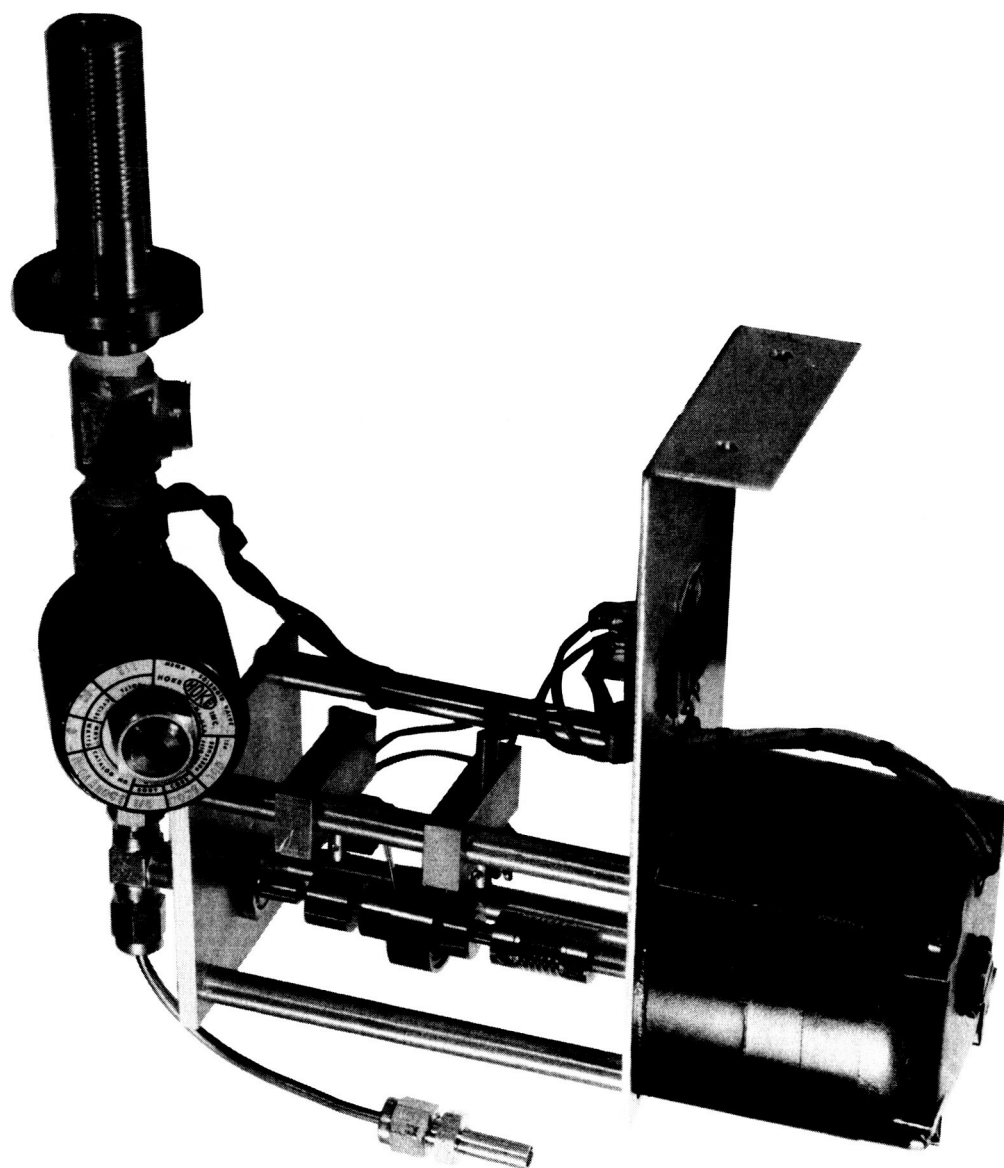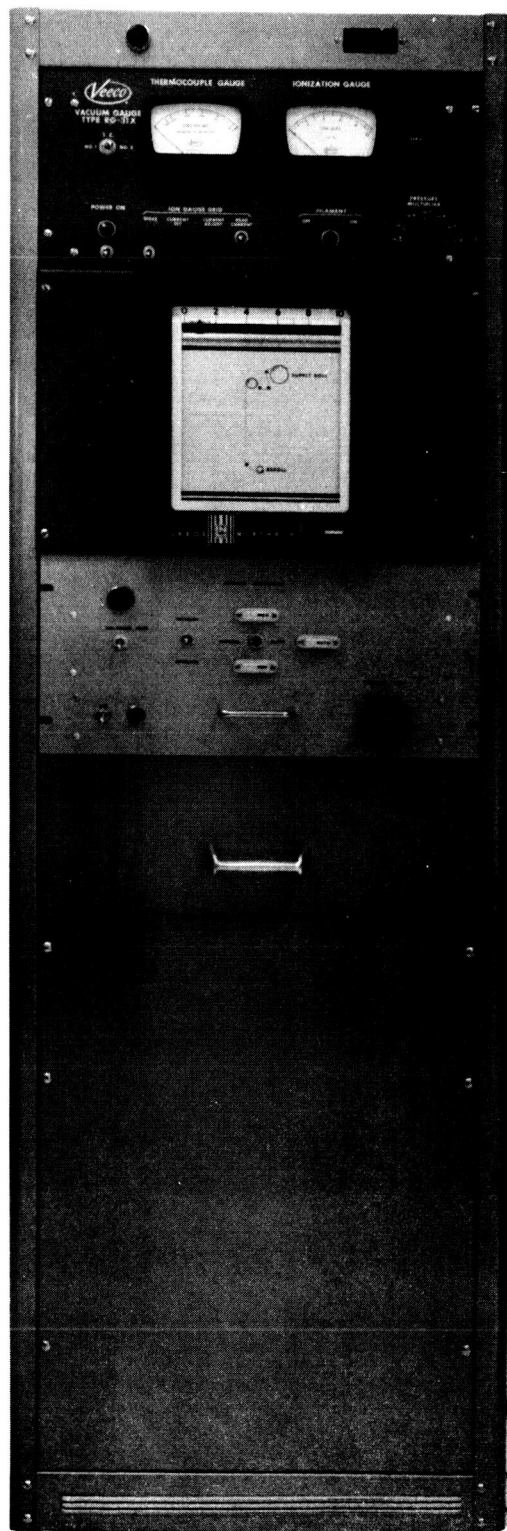A block diagram of the basic deposition-control system is shown in Figure C-1. The detector unit consists of a 9.35-megacycle, crystal-controlled transistor oscillator which is placed in the vacuum system in such a location that the crystal is adjacent to the substrate and receives a characteristic sample of the material(s).

The output of the sample oscillator, a nominally 9.35-megacycle RF signal, is fed through a coaxial cable into a transistor mixer, where the difference between the sample oscillator and a reference oscillator is obtained. This different frequency, which is typically 1 to 100 kc, is then converted by an analog frequency meter to a dc output current which is proportional to the difference frequency. As this difference frequency is proportional to deposited mass, the output of the frequency meter is proportional to the deposited mass. This signal is recorded as "Deposited Mass" on the strip-chart recorder.

An operational amplifier differentiator operates on the deposited mass signal and produces a mass deposition-rate signal, or simply, a deposition-rate signal. This voltage is recorded on the "Deposition Rate" chart recorder.

The rate signal is also fed to the rate programmer unit which consists of a variable attenuator network whose characteristics are established on the basis of the desired deposition rate. The attenuated rate signal is now compared to a reference voltage, and any difference or error signal is amplified by a servo amplifier. This resulting amplified error signal drives a servo motor which increases or decreases the voltage applied to the evaporator unit in a manner to reduce the error signal to zero and thus to correct the deposition-rate signal to the desired value.

Physical and Electrical Characteristics
of Deposition-Control System

Physical features. - The complete control system is housed in a 6-foot relay rack. A frequency meter, the reference oscillator and mixer,
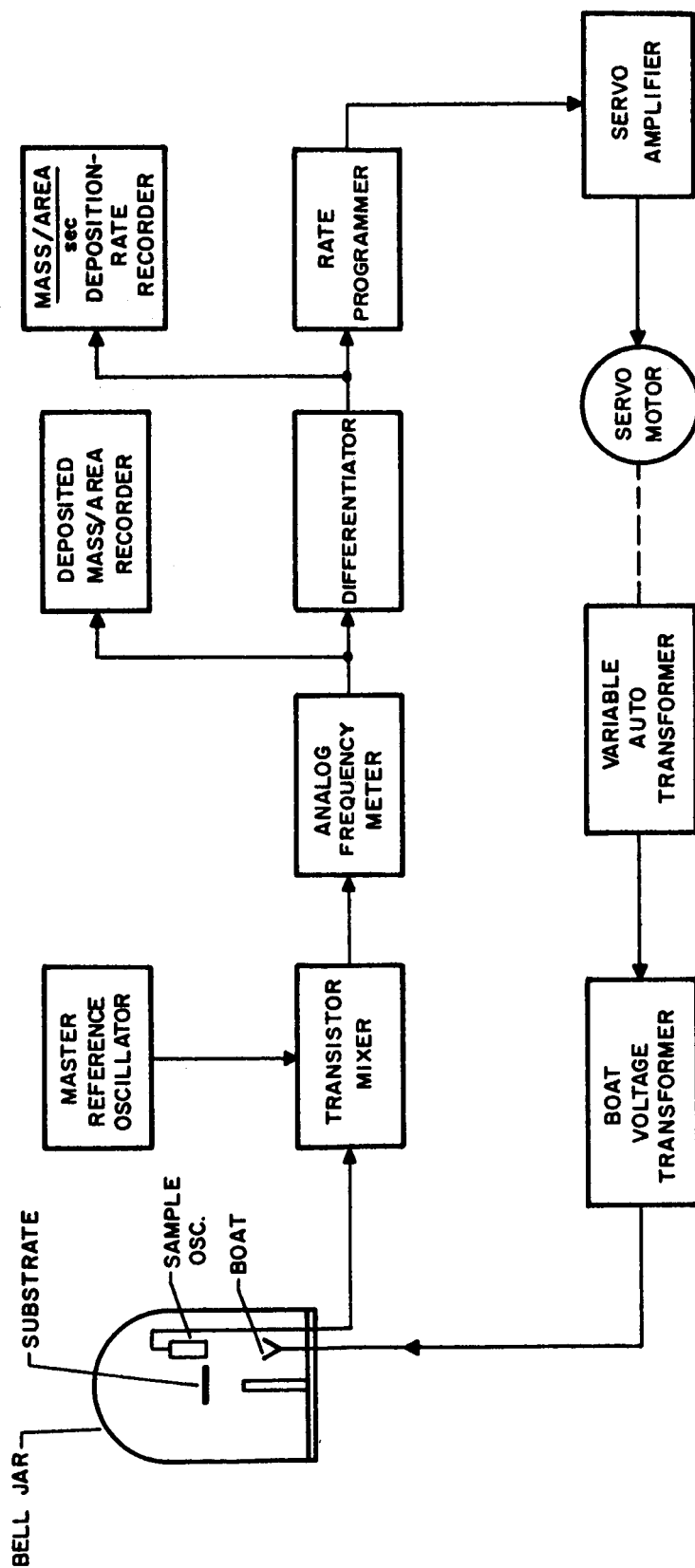
Figure C-1. R.Q.M. Deposition-Rate Controller, Block Diagram

the rate detector-programmer, the servo amplifier, and a power-control panel are mounted in the electronics rack along with the dual-channel recorder for mass and rate. Figure C-2 is a photo of the entire system less the dual-channel recorder.

Detailed description of the components. —

Sample Oscillator

The sample oscillator is a single-transistor, crystal-controlled oscillator constructed in a hermetically sealed enclosure that is installed in the vacuum system. The dc bias and ac output are handled by a single coaxial cable. Figure C-3 is a photograph of an unassembled unit, the schematic of which is shown in Figure C-4. A parts list for the sample oscillator is on the page following Figure C-4. The short-term stability of these oscillator units is better than one part in 10^7.

A 9.350-Mc fundamental frequency was chosen to obtain the desired sensitivity. The sensitivity of the crystal resonant frequency to mass loading is given by

$$\Delta f = - \frac{f^2}{N \rho_q} \frac{M}{A} \qquad 3.$$

where

Δf = frequency shift (cps)

f = fundamental frequency (9.350 Mc)

N = crystal resonant frequency coefficient (167 kc-cm)

ρ_q = mass density of quartz (2.65 grams cm^{-3})

M/A = mass per unit area of the deposit (grams cm^{-2})

Thus,

$$f = 2.00 \times 10^8 \frac{M}{A} \quad cps$$

and we can express the sensitivity as

$$S = 5.00 \ \mu grams \ cm^{-2} \ kc^{-1}$$

---

3. Sauerbrey, G., op. cit.
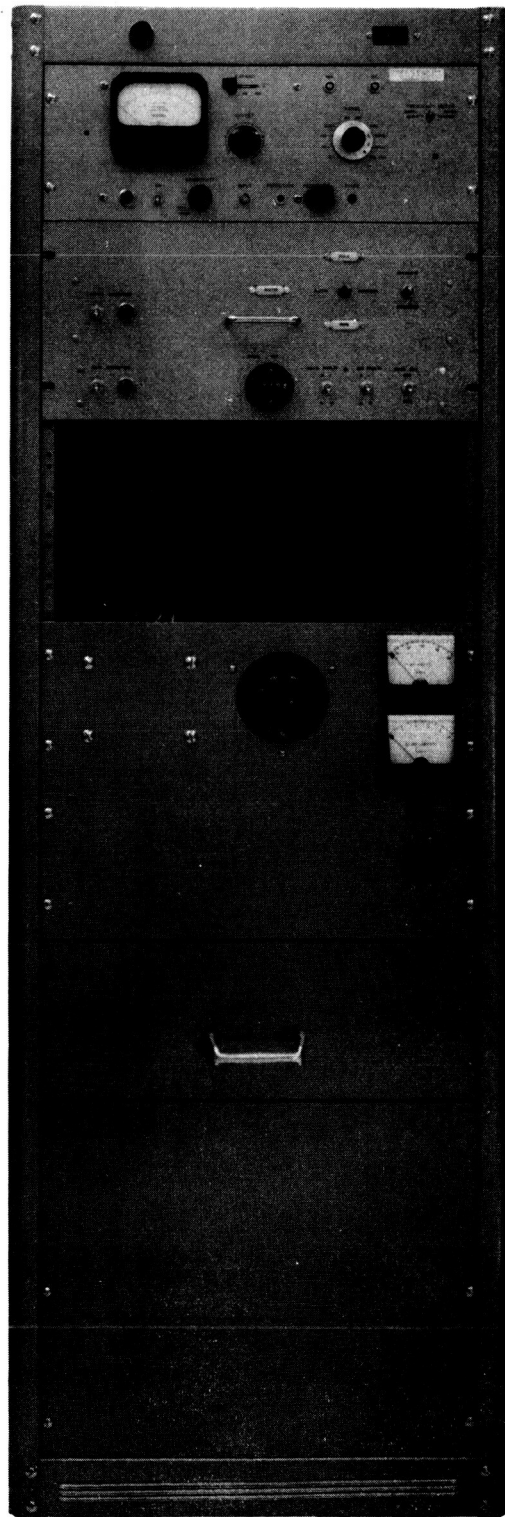
Figure C-2. Complete Control System
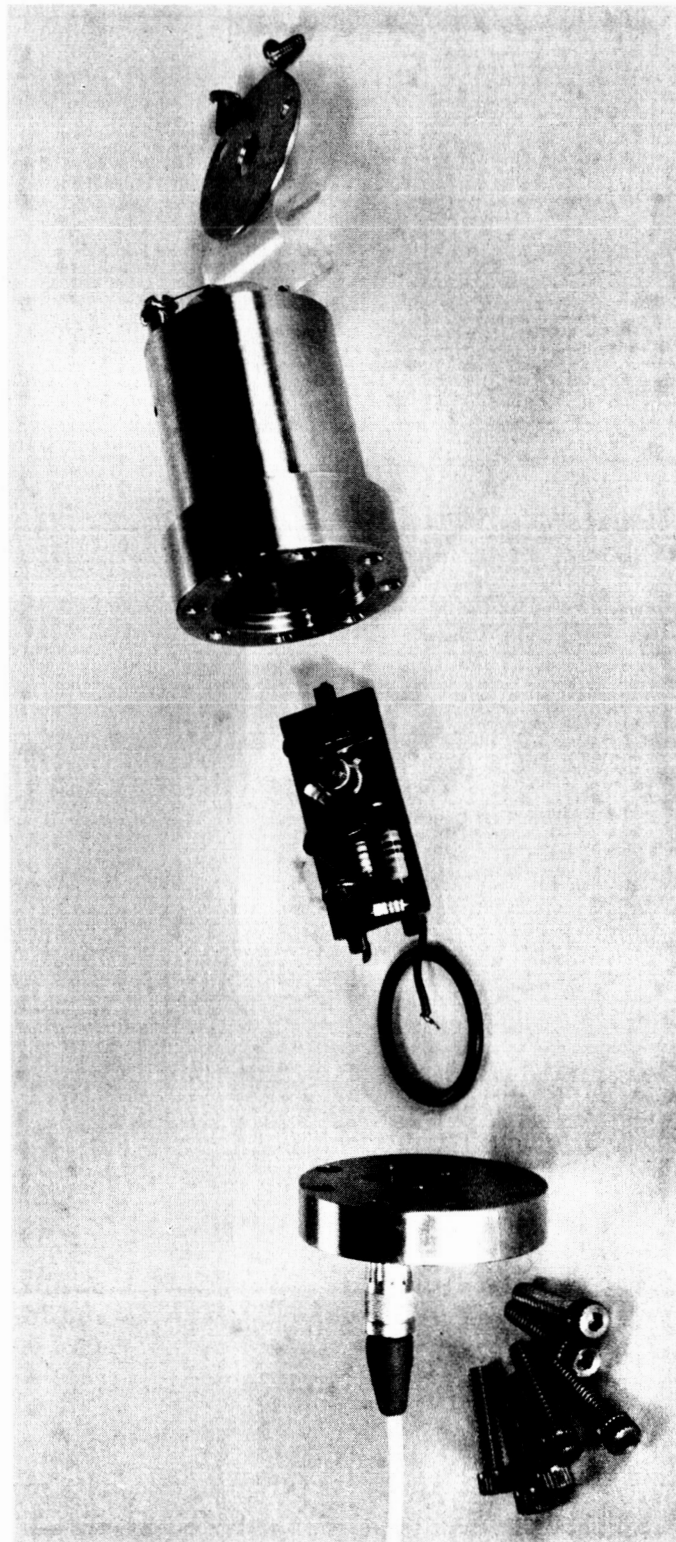
Figure C-3. Unassembled Sample Oscillator
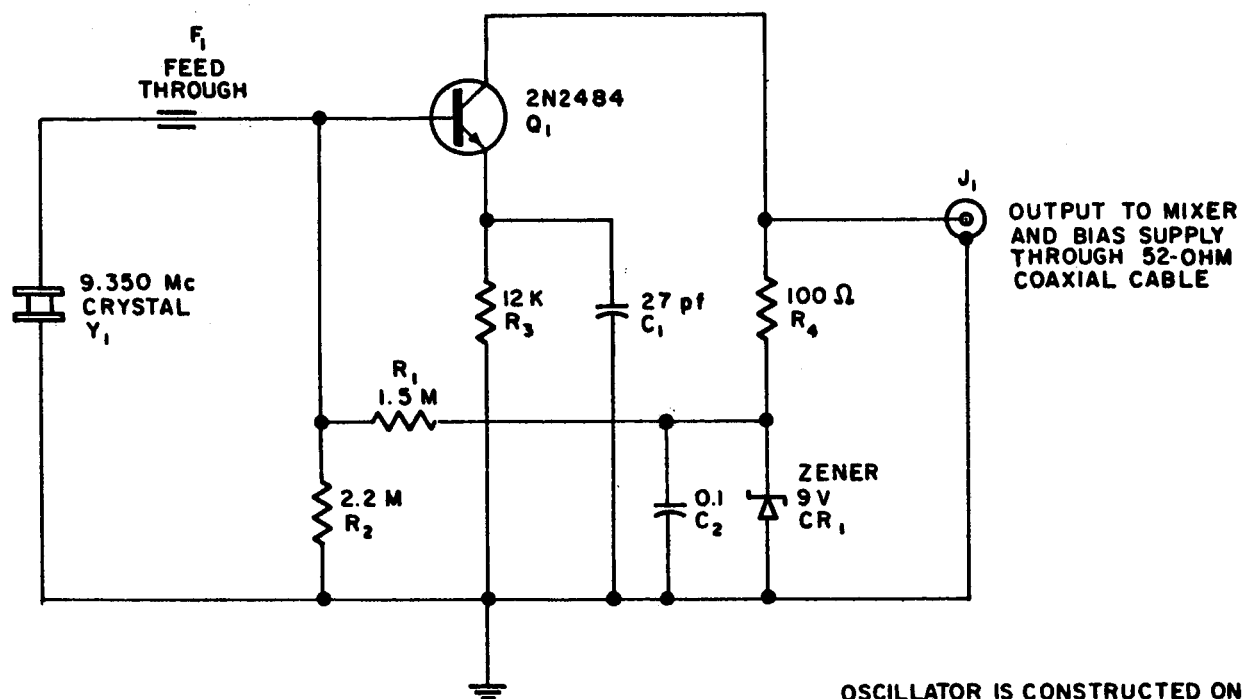
Figure C-4. Sample Oscillator, Schematic Diagram

Parts List for Sample Oscillator
(Figure C-4)

Symbol	Description
R_1	1.5 meg., 10%, 1/10 watt, carbon resistor,
R_2	2.2 meg., 10%, 1/10 watt, carbon resistor
R_3	12K, 10%, 1/10 watt, carbon resistor
R_4	100 ohm, 10%, 1/10 watt, carbon resistor
C_1	27 pf, mica capacitor
C_2	0.01 µf, 25 vdcw, disc capacitor
Q_1	Transistor, 2N2484
CR_1	Zener diode, 1N713
Y_1	9.350-Mc crystal, 0.005%, series resonant, Bliley No. BH6A
J_1	Microdot slide-on receptable, No. 51-158
F_1	Feed through, ceramic, No. 800A0720-1 Ceramaseal, Inc.

Figure C-5 is the circuit-board layout for the sample oscillator. The mechanical details of the oscillator housing are covered in Figures C-6, C-7, and C-8, and a completed assembly view is shown in Figure C-9.

The quartz crystal is mounted on the face end of the sample oscillator housing with two wire clips. Figure C-10 is a photograph of the oscillator unit with a crystal installed but with the crystal mask removed.

Reference oscillator and mixer

A transistorized master reference oscillator, buffer amplifier, and transistor mixer are located on a subchassis in the deposition-rate monitoring unit. A schematic of this subchassis is shown in Figure C-11; the parts list for Figure C-11 appears on the page following the schematic.

Analog frequency meter

A Hewlett-Packard 500 BR frequency meter provides the conversion from a difference-frequency signal to a dc current. The frequency-meter unit has been modified in the following way:

a. A hum-bucking network, consisting of a series resistance and capacitance, was connected from the secondary of the high-voltage transformer to ground. The component values are adjusted to minimize the residual 60 cps in the recorder output.

b. The gas reference tube in the power supply has been replaced by a semiconductor diode to improve the stability of the power supply.

c. The 6AL5 phototube bias rectifier was removed.

The net result of these changes is to reduce the 60-cycle hum in the output signal and to improve the stability. In addition, rear-mounted input and output connections are installed.

The frequency-meter output is always 1.0 ma for a full-scale reading; thus,

$$I_f = \frac{f_f}{f_o} \times I_o \text{ amps}$$
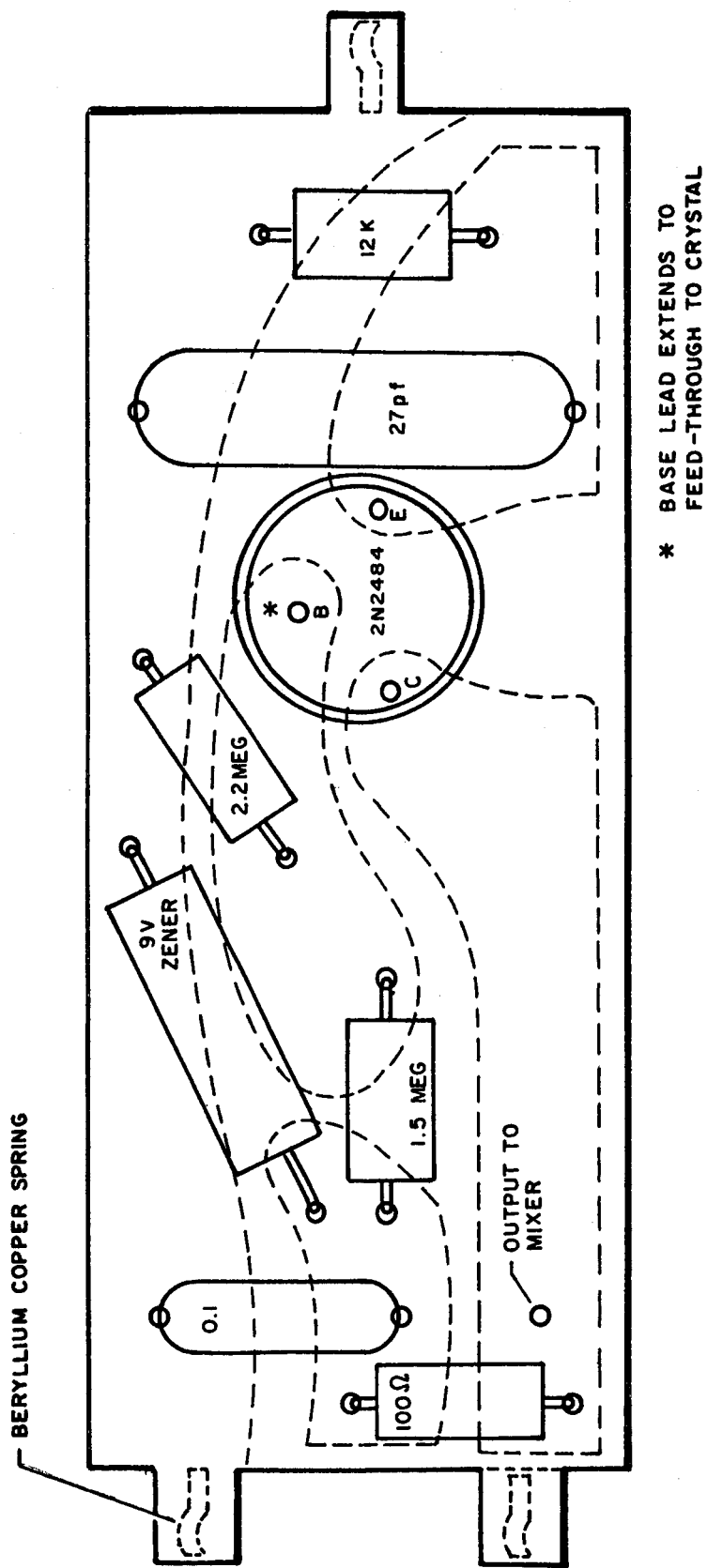
Figure C-5. Circuit Board for Quartz Crystal Microscale Sample Oscillator

41

Figure C-6. Oscillator Housing

42

Figure C-7. End Plate

.218 DIA. DRILL THRU .250 DIA $^{+.005}_{-.000}$ x .020 DP $^{+.005}_{-.000}$
C'BORE THIS SIDE

45°

$^{+.005}_{}$ .070

11 $^{+.005}_{}$ .64

(8) .120 DIA HOLES
EQUALLY SPACED ON
A 1.000 DIA. B.C. ±.002

.314 ±.005

.157 ±.002

(2) .125 DIA. END MILL
.125 DP. ±.005 FAR SIDE

.250 ±.010

32

1.187 $^{+.005}_{-.000}$

MAT'L - 316 ST. ST'L
BREAK SHARP EDGES
(1) REQ'D.

43

A

4-40 THD

1/16 WIDE x .080 DIA.
UNDER CUT

±.005
.281

±.005
.562

SLOT FOR
SCREW DRIVER

2-56 TAP x 3/16 DP
MAX. DEPTH OF TAP DRILL 1/4

STAND OFF

MAT'L- 316 ST. ST'L (2 REQ'D)

B

(2) .125 DIA. HOLES
AS SHOWN ON
.781±.002 DIA. B.C.

45°

.896 +.000

+.002
.220 DIA.
(TO SUIT
XTAL USED)

CRYSTAL MASK

MAT'L 316 ST. ST'L .032 THK (I REQ'D)

C

6-32 TAP

LOCKING COLLAR

MAT'L 316 ST. ST'L (I REQ'D)

+.005
-.000
1.195 DIA.

15/16 DIA.

Figure C-8. Details

44

HERMETIC SEAL COAX
FEED-THROUGH MICRODOT
51-158 (I REQ'D)

SOLDER (PRESSURE
TIGHT)

S.S. SOC. HD. SCREW #4-40×1/2 (8 REQ'D)

END PLATE WF-2 (I REQ'D)

O-RING PARKER VITON #2-17 (I REQ'D)

HOUSING WF-1 (I REQ'D)

STANDOFF WF-3-A (2 REQ'D)

SPRINGS SOLDERED
TO STANDOFF & FEED-
THROUGH (2 REQ'D)

.265 +.010 -.000

CUT TERMINAL TO
THIS DIMENSION
AFTER ASS'Y.

TERMINAL "CERAMASEAL"
CAT. # 800A0720-1

SOLDER (PRESSURE TIGHT)

Figure C-9. Ultrahigh-Vacuum Crystal Oscillator

45

Figure C-10. Assembled Sample Oscillator

46

Figure C-11. Master Reference Oscillator and Mixer

47

Parts List for Master Reference Oscillator and Mixer
(Figure C-11)

Symbol	Description
R_1	220K, 10%, 1/2 watt, carbon resistor
R_2	12K, 10%, 1/2 watt, carbon resistor
R_3, R_5	5.6K, 10%, 1/2 watt, carbon resistor
R_4, R_7, R_8	39 ohms, 10%, 1/2 watt, carbon resistor
R_6	470 ohms, 10%, 1/2 watt, carbon resistor
R_9	8.2K, 10%, 1/2 watt, carbon resistor
R_{10}	220 ohms, 10%, 1/2 watt, carbon resistor
R_{11}	10K, 10%, 1/2 watt, carbon resistor
R_{12}	56 ohms, 10%, 1/2 watt, carbon resistor
R_{13}	1.2K, 10%, 1/2 watt, carbon resistor
C_1, C_2, C_6	1.0 µf, disc capacitor, 25 vdcw
C_3	330 pf, mica capacitor
C_4, C_8	0.01 µf disc capacitor, 25 vdcw
C_5	27 pf, mica capacitor
C_7	150 pf, mica capacitor
C_9	0.003 µf disc capacitor, 25 vdcw
C_{10}*	10 pf, mica capacitor

* Select for proper reference frequency.

Parts List for Master Reference Oscillator and Mixer (Cont)
(Figure C-11)

Symbol	Description
Q_1	Transistor, 2N2484
Q_2, Q_3	Transistor, 2N708
Y_1	9.350-Mc crystal, 0.005%, series resonant, Bliley No. BC61A
L_1	10 μh, molded RF choke
J_1, J_2	BNC, UG-1094/U

where

f_o is full-scale reading

f_f is frequency-meter reading

I_o is 1 ma.

The frequency shift is proportional to the mass deposit

$$\Delta f \; = \; \frac{\sigma}{S}$$

where

σ is surface mass in μgrams cm^{-2}

S is sensitivity in μgrams cm^{-2} $cycle^{-1}$

In using this system, one can neglect any initial frequency-meter reading as being equivalent to a previous deposited mass;

$$\therefore \; f_f \; = \; \frac{\sigma}{S}$$

where

f_f = frequency-meter reading.

$$I_f \; = \; \frac{f_f}{f_o} \; I_o \; \text{from frequency meter}$$

$$I_f \; = \; \frac{\sigma}{S} \; \frac{I_o}{f_o}$$

Mass recorder

The mass recorder voltage is obtained from the voltage drop across R_3, Figure C-12; the parts list for Figure C-12 appears on the page following the schematic.

* SELECTED TO PROVIDE PROPER DAMPING

Figure C-12. Rate Detector

51

Parts List for Rate Detector
(Figure C-12)

Symbol	Description
R_1	400 ohms, 1%, film resistor, 1/2 watt
R_2	990 ohms, 0.1%, film resistor, 1/2 watt
R_3	10 ohms, 0.1%, film resistor, 1/2 watt
R_4	56K, 1%, film resistor, 1/2 watt
R_5	4.7 meg., 1%, film resistor, 1/2 watt
R_6	470K, 1%, film resistor, 1/2 watt
R_7	100K, potentiometer, carbon, 2-watt
R_8	1 meg., potentiometer, carbon, 2-watt
R_9	2.5K, 0.1%, film resistor, 1/2 watt
R_{10}	50K, 5%, potentiometer, 10-turn W.W., Borg No. 2201B
R_{11}	4.5K, 0.1%, film resistor, 1/2 watt
R_{12}	500 ohms, 0.1%, film resistor, 1/2 watt
R_{13}	100 ohms, potentiometer, carbon, 2 watt
R_{14}	150 ohms, 1%, film resistor, 1/2 watt
R_{15}	270K, 1%, film resistor, 1/2 watt
R_{16}	27K, 1%, film resistor, 1/2 watt
R_{17}	320K, 1%, film resistor, 1/2 watt
C_1, C_2, C_3	105 µf 15 vdc, tantalum, electrolytic, Sprague No. 107X901OR2
C_4	1.0 µf 200 vdc, molded paper capacitor

Parts List for Rate Detector (Cont)
(Figure C-12)

Symbol	Description
C_5	0.1 µf 200 vdc, molder paper capacitor
C_6	Selected to provide proper damping; typically 50-100 µf electrolytic
C_7, C_8, C_9, C_{10}	0.001 µf feed-through capacitor, Centralab No. FT
J_1	BNC UG-1094/U
J_2, J_3, J_4	Connector, audio, female, Amphenol No. 91-856
D_1, D_2	Diode, FD100
S_1, S_3, S_4	Switch, SPDT, Cutler-Hammer No. 8836K4
S_2	Switch, DPDT, Cutler-Hammer No. 8837K4
P_2	Differential operational amplifier, Philbrick Research, Inc., P2A
F_1	Fuse, 2 ampere
G_{p1}	Parallel ground plug, male
Pl_1	Pilot light, Dialco No. 931
QM_1, QM_2	Power supply, 16v, 100 ma, Technipower No. M-158-0.100

Thus, mass recorder voltage

$$V_\sigma = I_f R_3 = \frac{\sigma}{S} \frac{I_o}{f_o} R_3$$

$$I_o = 10^{-3} \text{ amps}$$

$$S = 5 \text{ } \mu\text{grams cm}^{-2} \text{ kc}^{-1}$$

$$R = 10 \text{ ohms}$$

$$f_o = \text{frequency-meter setting in cycles}$$

$$V_\sigma = \sigma \left[\frac{10^3}{f_o} \times \frac{10^{-3} \times 10}{5 \times} \right] = \sigma \frac{2}{f_o}$$

or

$$\sigma = \frac{V_\sigma f_o}{2} \text{ in } \mu\text{grams cm}^{-2}$$

The differentiator or rate detector

The differentiator circuit shown in Figure C-12 uses an operational amplifier to obtain stable, low-noise differentiation of the mass versus time signal, and provides a low-impedance output to drive the rate recorder and the rate programmer network. The circuit is a variation of the one suggested by the G. A. Philbrick Company. The output of the differentiator is a dc voltage that is proportional to the rate of change of the deposited mass signal and which is proportional to the deposition rate. A deposition-rate multiplier is included as part of the differentiator. This range switching is achieved by varying the feedback resistor. The rate multiplication factor is 10 to 1. The capability of changing the sensitivity of the differentiator in this manner increases the versatility of the deposition control system by allowing one to choose the optimum frequency-meter setting for the desired deposited mass and still obtain optimum rate-detector performance. The output of the rate detector (differentiator) is fed directly to the deposition recorder. The magnitude of this signal is proportional to the deposition

54

rate and may range from 0 to 100 millivolts. The actual calibration of this signal is dependent upon the frequency-meter settings and the differentiator multiplier setting.

The following are differentiator circuit equations (ref. Figure C-12):

The differnetiator output is

$$e_d = \frac{dV_{in}}{dt} R_f C_3$$

where

R_f is feedback resistor, either R_5 or R_6, depending on differentiator multiplier (S_3) setting.

V_{in} is the input voltage produced by the frequency-meter current flowing through R_2 and R_3

$$V_{in} = I_f (R_2 + R_3)$$

$$\frac{dV_{in}}{dt} = \frac{d I_f}{dt} (R_2 + R_3)$$

Therefore,

$$e_d = \frac{d I_f}{dt} (R_2 + R_3) R_f C_3$$

as before

$$I_f = \frac{\sigma}{S} \frac{I_o}{f_o}$$

as only σ is time variant

$$\frac{d I_f}{dt} \quad \frac{d\sigma}{dt} \quad \frac{I_o}{Sf_o}$$

$\frac{d\sigma}{dt}$ is deposition rate (μgams cm^{-2} sec^{-1})

55

define

$$\frac{d\sigma}{dt} \quad D \equiv \mu grams\ cm^{-2}\ sec^{-1}$$

$$\frac{dI_f}{dt} = D\frac{I_o}{Sf_o}$$

Therefore,

$$e_d = D\frac{I_o}{Sf_o}(R_2 + R_3)\ R_f\ C_3$$

Substituting

$$I_o = 10^{-3}\ amps\ full\ scale$$

$$S = 5\ \mu grams\ cm^{-2}\ kc^{-1}$$

$$R_2 = 990\ ohms$$

$$R_3 = 10\ ohms$$

$$C_3 = 105\ \mu f\ nominal$$

$$R_{f1} = R_5 + R_8 = 4.76 \times 10^6\ ohms\ nominal \quad x\ 1\ on\ Diff.\ Mult.$$

or

$$R_{f10} = R_6 + R_7 = 4.76 \times 10^5\ ohms\ nominal \quad x\ 10\ on\ Diff.\ Mult.$$

In calibration $\frac{R_{f1}}{R_{f10}} = 10/1$ and

R_{f1} and R_{f2} are adjusted to produce the desired calibration allowing for the variation in C_3 ($R_{f1}\ C_3 = 500\ sec$).

Therefore,

$$e_d = \frac{D\ 10^{-3}}{f_o\ 5\ 10^{-3}} \left(1,000 \left(\frac{4.76 \times 10^{-6}}{M_D} \right) (10^5)\ (10^{-6}) \right)$$

where M_D is setting of S_3, 1 or 10

$$e_d = \frac{D\ 5 \times 10^5}{f_o\ 5\ M_D} \quad \text{volts } \mu\text{grams}^{-1}\ \text{cm}^{-2}\ \text{sec}^{-1}$$

$$e_d = \frac{D}{f_o\ M_D} \times 10^5$$

or

$$D = e_d \frac{f_o\ M_D}{10^5} \quad \mu\text{grams cm}^{-2}\ \text{sec}^{-1}$$

Rate recorder

The output (e_d) of the differentiator is fed directly to the rate recorder. Thus, the deposition rate can be read directly from the recorder as

$$D = e_d \left[\frac{f_o\ M_D}{10^5} \right] \quad \mu\text{grams cm}^{-2}\ \text{sec}^{-1}$$

Rate programmer unit

The rate programming unit provides the means for achieving the desired deposition rate. Fundamentally, the rate programmer is a simple resistive divider. The input voltage to this divider is proportional to the deposition rate. The output voltage, a fraction of the input voltage, is compared to a reference voltage by the servo amplifier and held constant. This produces a transfer function as shown below (cf. Figure C-13).

e_r = output voltage = reference voltage = a constant

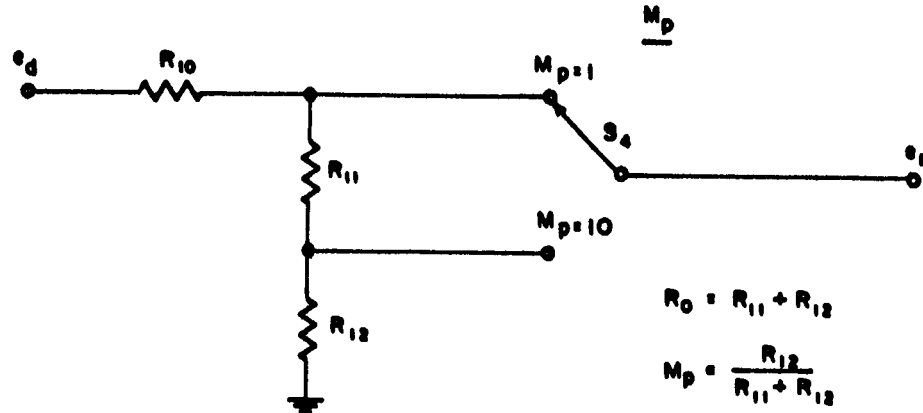e_d = deposition-rate signal from differnetiator

Figure C-13.   Rate Programmer Network

For this network let

$$N = \left(1 + \frac{R_1}{R_o}\right) \quad ; \quad \text{therefore,} \quad \frac{e_d}{e_r} = M_p N$$

R_{10} can now be changed to provide a linear variation of the deposition rate if

$$R_{10max} = 10 R_o$$

and

$$R_{10} = R_{10max} X, \quad 0 \le X \le 1$$

58

Then,

$$N = 1 + \frac{X R_{10max}}{R_o} = 1 + X \frac{10R_o}{R_o} = 1 + 10X$$

If R_{10} is fitted with a dial which reads 1.0 when $R = 0$ ($X = 0$) and 11 for $R_{10} = R_{10max}$ ($X = 1$) then N is dial reading. A variation of 1.0 to 11 in N obtained with a direct-reading dial on the 10-turn rheostat used for R_{10}.

Combining the rate programmer and the differentiator equations,

$$M_p N = \frac{e_d}{e}$$

and

$$e_d = \frac{D}{f_o M_D} \times 10^5$$

$$D = \frac{N e_r f_o M_D M_p}{10^5}$$

e_r is set to 0.010 volt during calibration.

Therefore,

$$D = N f_o M_D M_p \times 10^{-7} \ \mu grams \ cm^{-2} \ sec^{-1}$$

This is the defining equation of the rate-control system.

Servo Amplifier

The servo amplifier is a Minneapolis-Honeywell unit containing a chopper for converting the dc signals to 60-cycle ac suitable for driving the servo motor. This is a standard unit with no modifications. A damping network as shown in Figure C-12 ($C_6 R_{15}$) suppresses the tendency of the servo system to oscillate. The time constant of the damping network must be adjusted to an optimum value that is dependent upon the physical construction of the evaporator unit. The wiring of the servo amplifier and its controls is shown in Figure C-14. The parts list for this unit is on the page following the illustration.
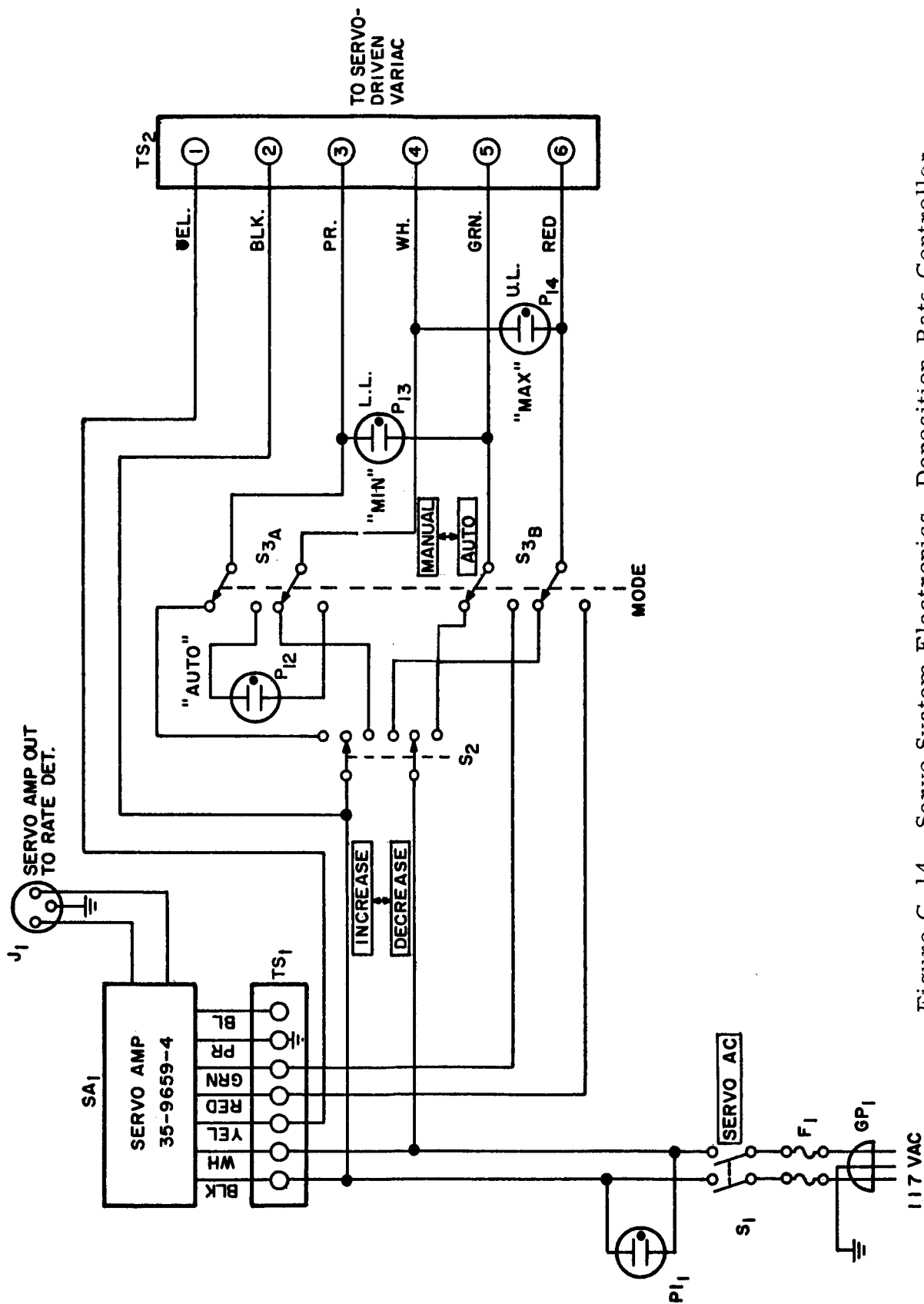
Figure C-14. Servo System Electronics, Deposition-Rate Controller

60

Deposition-Rate Controller Servo System
(Figure C-14)

Symbol	Description
G_{P1}	Parallel ground plug, male
F_1	Fuse, 1 ampere, slow-blow
Pl_1	Pilot light, Dialight No. 931
S_1	Toggle switch, ac, Cutler-Hammer No. 8823K5
SA_1	Servo amplifier, Honeywell No. 35-9659-4
J_1	Connector, audio, male, Amphenol 91-853
T_{S1}	Terminal strip, Cinch-Jones, 7-terminal
Pl_1, Pl_2, Pl_3	Indicator lights, Drake No. 110-022 (Hi Brite)
S_2	Switch, DPDT, Cutler-Hammer No. 8834K5
S_{3AB}	Switch, 4PDT, Cutler-Hammer No. 8838K4
T_{S2}	Terminal strip, Cinch-Jones, 6-terminal

The servo-system gain is adjusted with the internal gain control in the servo amplifier. The gain must be high enough to get the desired accuracy in rate. However, as gain is increased, the system may oscillate. The damping network suppresses this oscillation to some extent, allowing higher gain before oscillation begins. The optimum gain is just below the oscillation point and must be set experimentally. Usually, there will be a wide range of gain that will satisfy the two requirements of small error (high gain) and no oscillation (low gain).

Mounted adjacent to the servo amplifier on the front panel of the rate-control electronics chassis are the controls for selecting manual or automatic variation in evaporator voltage and for operating the servo variac with a manual switch. These controls are used to set up the initial condition of evaporator voltage.

The rate detector, reference oscillator and mixer, rate programmer, servo amplifier, and motor variac controls comprise the rate controller electronics and are mounted on a single chassis. Figures C-15, C-16, and C-17 are photographs of the assembled electronics.

Power control panel

The Power Control Panel houses the servo driven variac, a manual range setting variac and a transformer which are connected to provide the desired range of voltage to the evaporator unit. Figure C-18 is a schematic of the power panel and the following page is the parts list; Figures C-19 and C-20 are photographs of the assembled panel.
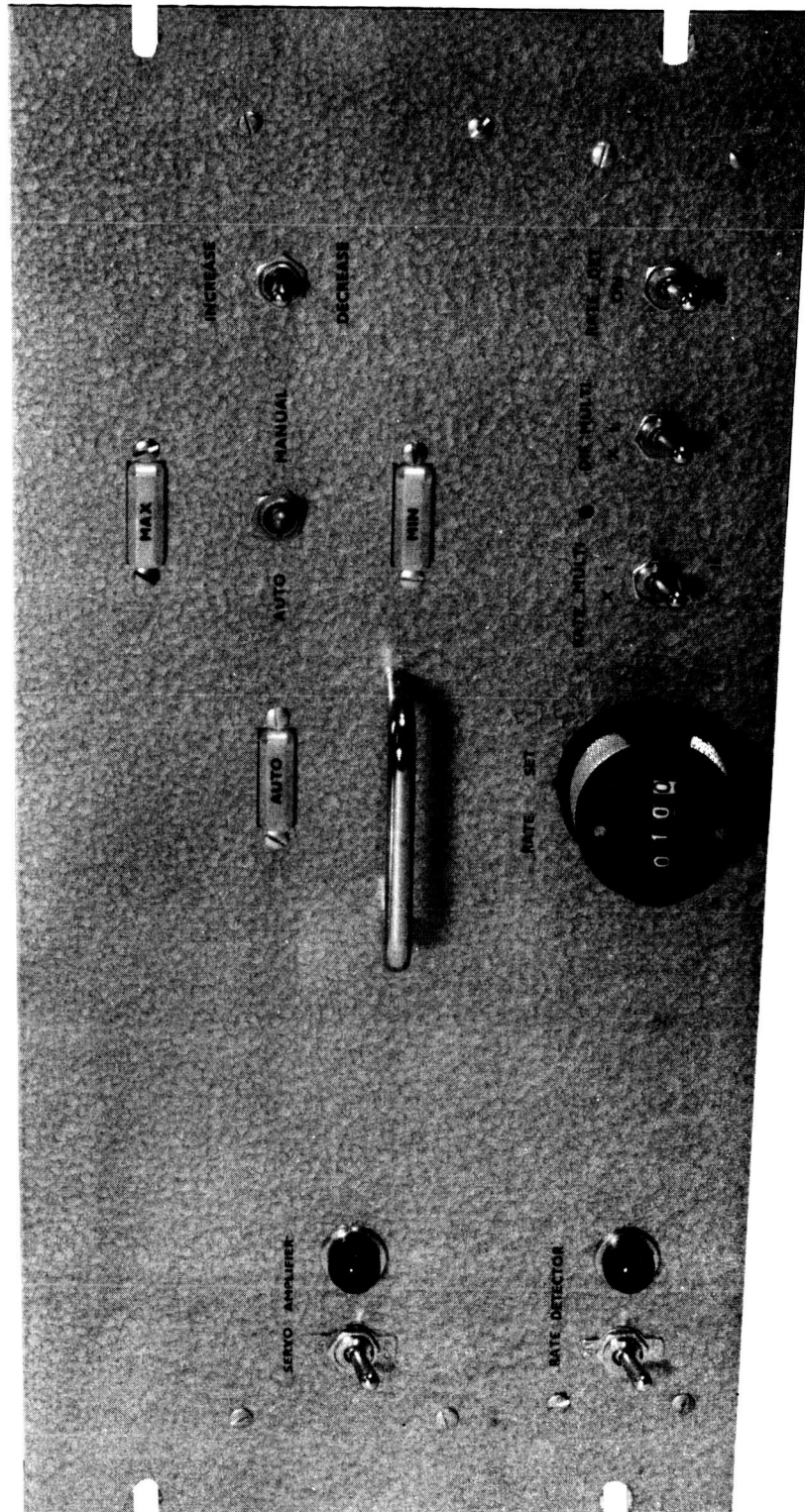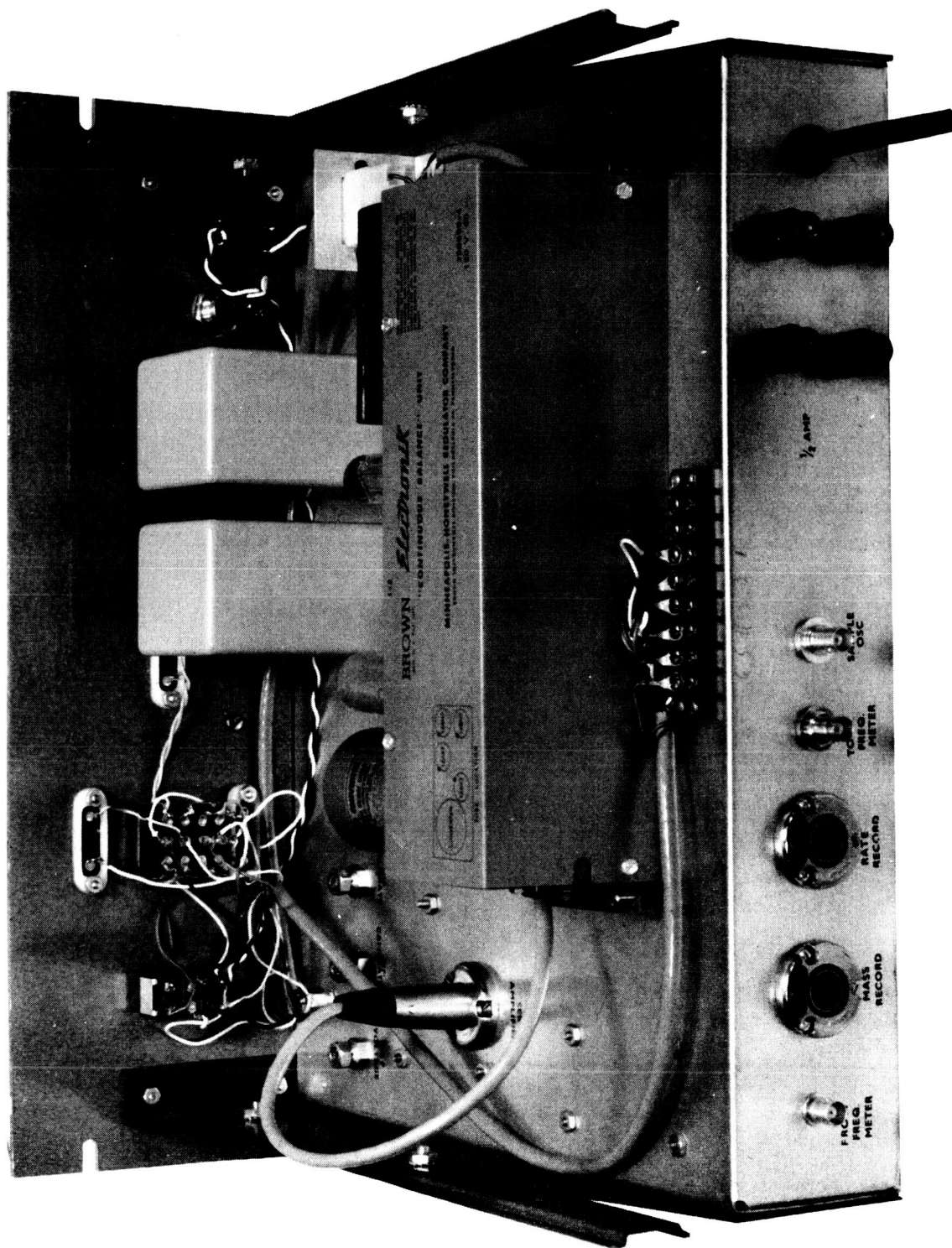
Figure C-15. Rate Controller, Front View
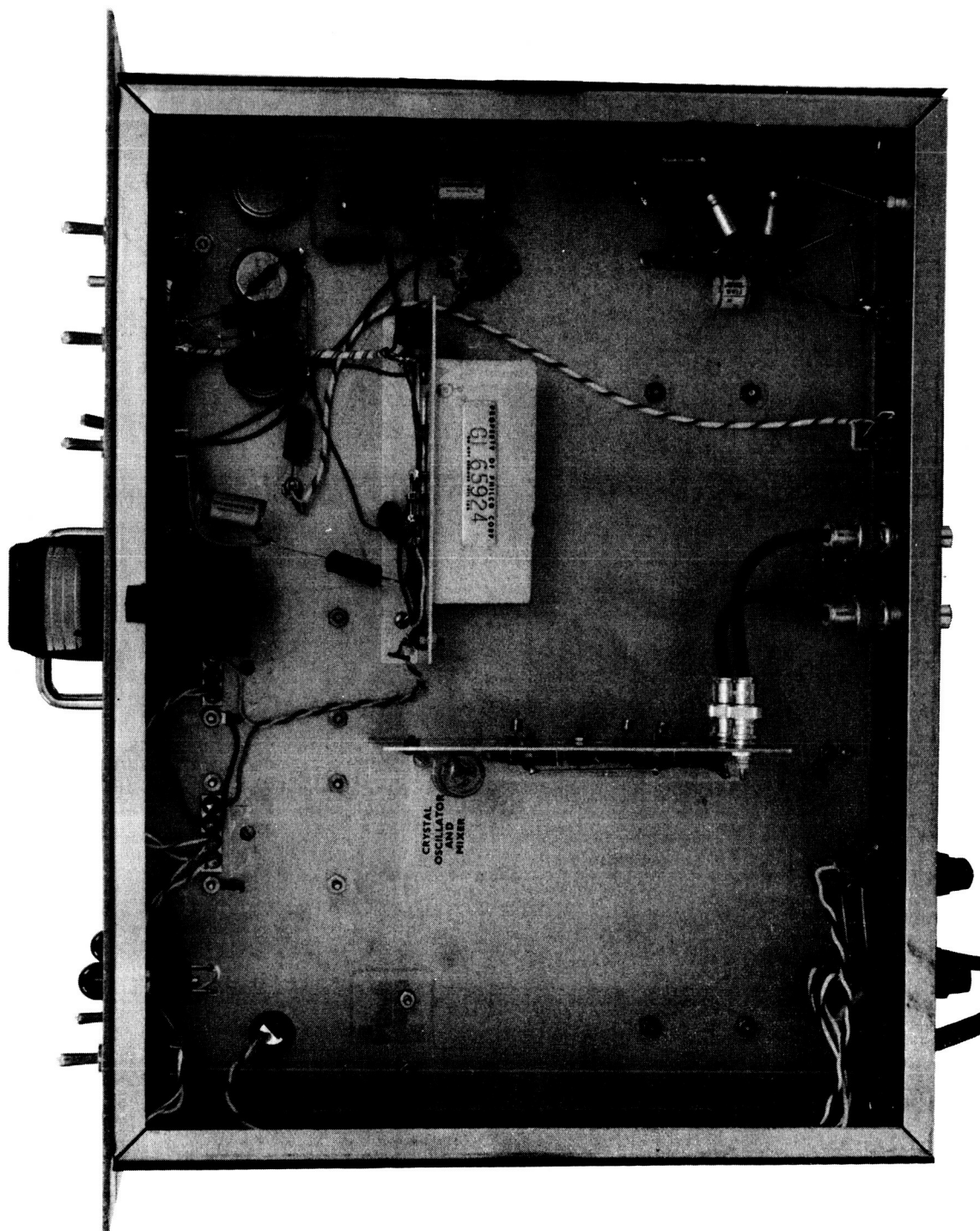
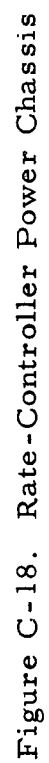Figure C-16. Rate Controller, Rear View

CRYSTAL
OSCILLATOR
AND
MIXER

PROPERTY OF PHILCO CORP.
CI 65924

Figure C-17. Rate Controller, Bottom View

Figure C-18. Rate-Controller Power Chassis

Parts List for Rate Controller Power Chassis
(Figure C-18)

| Symbol	Description
CB_1	Circuit breaker, 10-ampere, Wood Electric No. 190-210-101
Pl_1	Pilot light, Dialight No. 514001-211
VM_1	Variac, motor-driven, General Radio No. W10D128CKM
V_1	Variac, Superior Electric Co., No. 116U
M_1	Meter, 150 vac, 3-1/2 inch, Simpson No. 1357
M_2	Meter, select to match evaporator voltage
T_1	Transformer, multiltap, high current, sec 12 amperes, 2 windings, 18 volts
T_{S1}	Terminal strip, Cinch-Jones No. 3-140Y
T_{S2}	Terminal strip, Cinch-Jones No. 3-140Y
G_{P1}	Parallel ground plug, male
G_{P2}	Connector, chassis, 3-wire ac, male
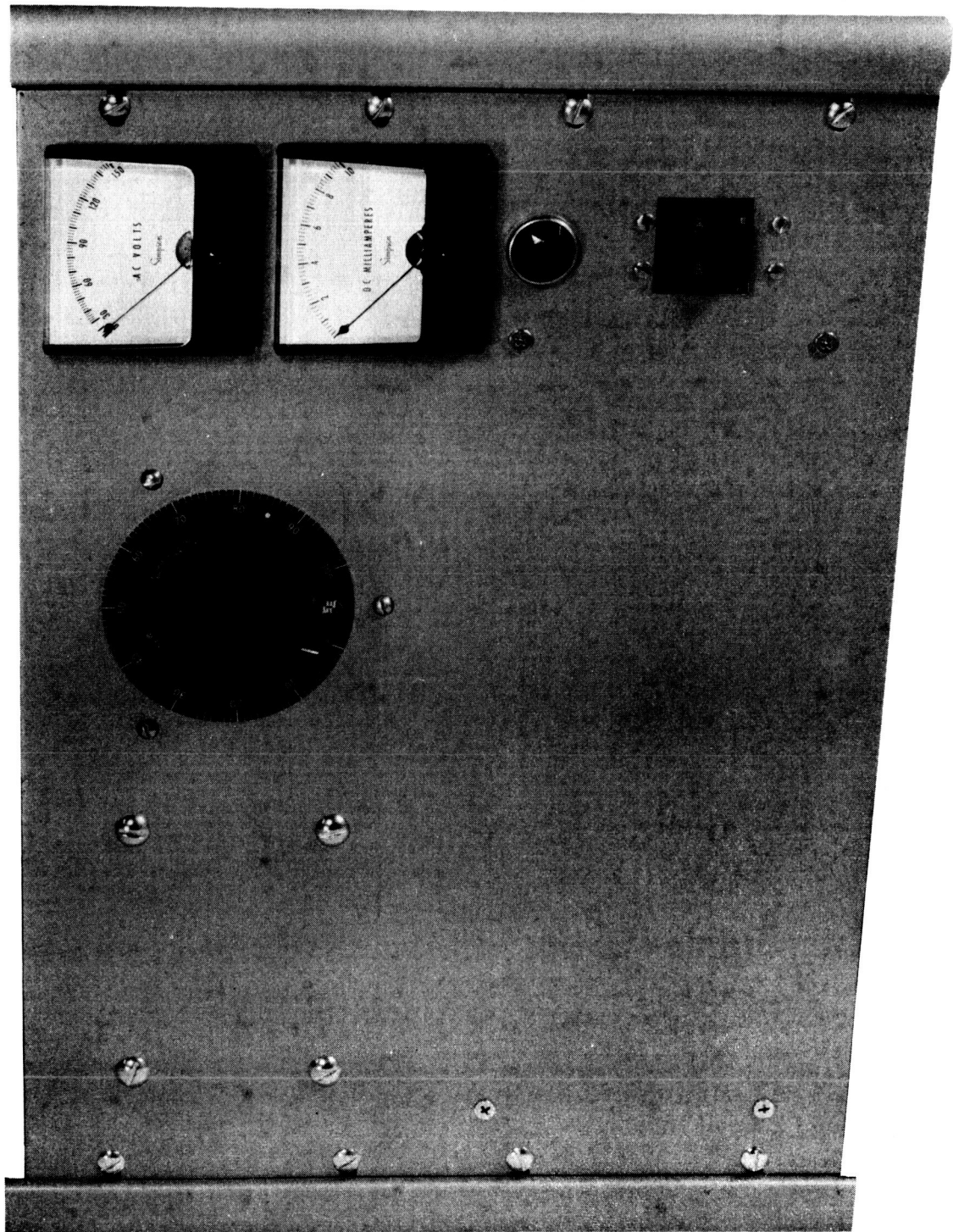
Figure C-19. Power Panel, Front View
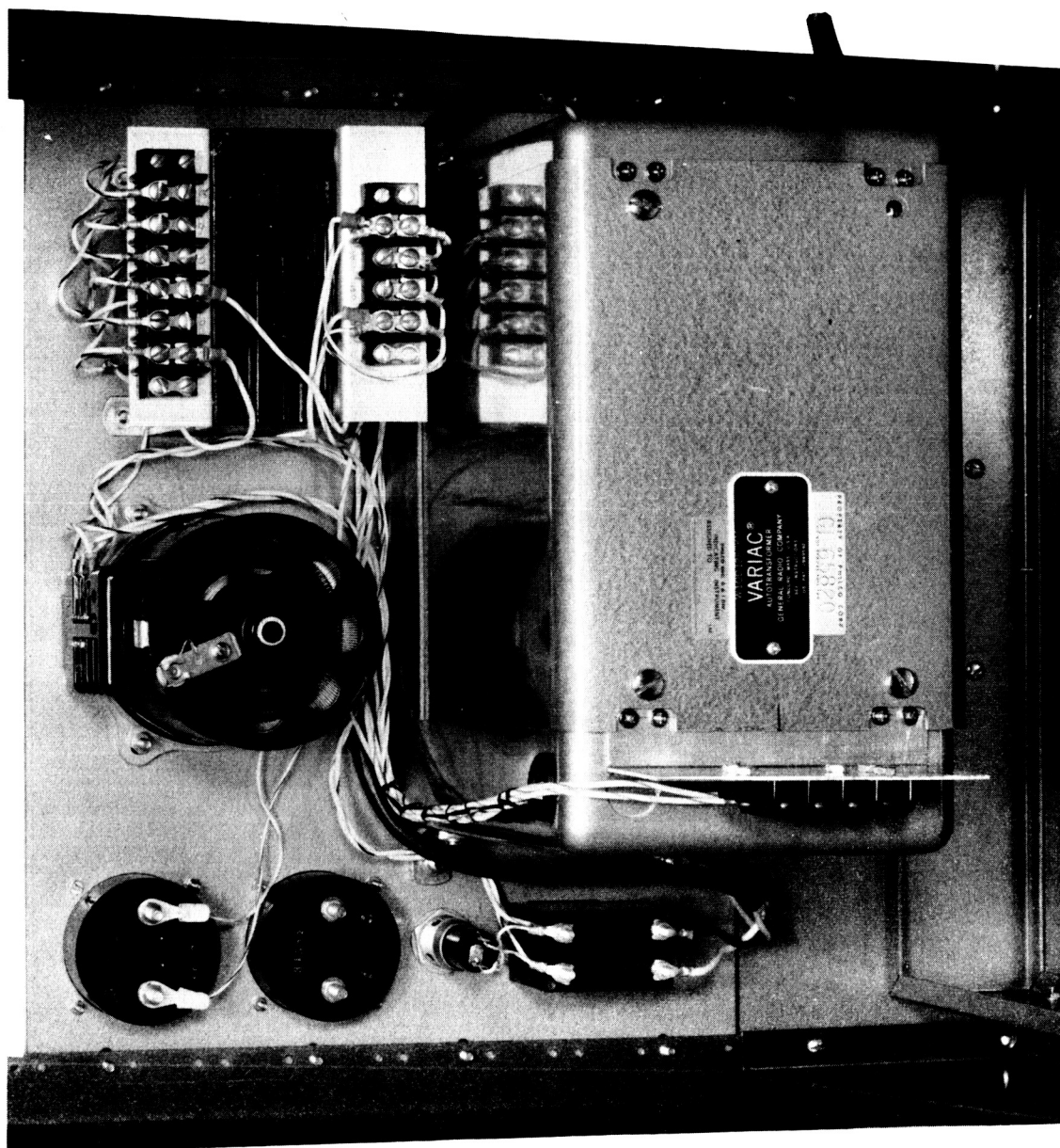
Figure C-20. Power Panel, Rear View

REFERENCES

1. Miles and Smith, "The Formation of Metal Oxide Films Using Gaseous and Solid Electrolytes," submitted to J. Electrochem. Soc.

2. Da Silva, E. M. and White, P., "Electrical Properties of Evaporated Aluminum Oxide Films," J. Electrochemical Soc., Vol. 109, No. 1, January 1962, p. 12.

3. Spratt, J. P., Final Report, Thin Film Active Devices, Contract No. DA-49-186-ORD-1056.

4. Kennard, E. H., Kinetic Theory of Gases, McGraw-Hill, New York.

5. Sauerbrey, G., Z. Physik, 155, 206, 1959.

6. Warner, A. W. and Stockridge, C. D., "Mass and Thermal Measurement With Resonating Crystalline Quartz," Vacuum Microbalance Techniques, Plenum Press, Vol. 2, 1961 pp. 71 to 92.

7. Berhardt and Love, Vac. Sys. Trans. p. 87, 1960; also Vacuum, pp. 1 to 9, Vol. 12, January-February 1962.

ABSTRACT

This report describes experimental and theoretical work on two areas of study regarding the production of radiation-resistant thin-film active devices.

An experimental and theoretical program is being pursued to determine the mean free path of energetic electrons obtained from a specially designed electron gun and injected into the metal surface-barrier contact of a metal semiconductor diode.

Aluminum oxide obtained by evaporating aluminum in a low-pressure oxygen environment will be studied with regard to electrical properties pertinent to the production of thin-film devices. The resistivity of the aluminum oxide can be controlled by variation in the oxygen pressure and deposition rate between the limits imposed by pure aluminum and pure aluminum oxide.